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SOME FACTORS INFLUENCING THE EXPERIMENTAL BAKING TEST

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Altho the value of the experimental baking test is accepted with different degrees of enthusiasm both in the profession and among the trade, it remains the most satisfactory means of determining flour quality and serves as the guide to mill control, bakery purchasing, and commercial laboratory operation.

Naturally there is some question as to the relative value of the experimental baking test, owing to the many methods of conducting it as well as to the formulas employed. The interpretations of results are also at variance, represented on the one hand by those who prefer to establish optimum conditions for each sample under test, and on the other hand by those who prefer to establish rigid conditions, holding the flour only as the variable.

The differences in interpretations led to the undertaking of a series of test bakings in an effort to establish certain definite data relating to the effect of the more common variations.

A well conditioned patent flour milled from hard winter wheat was selected for the work in all the series unless otherwise indicated. The analysis of the flour is given in Table I.

TABLE I

ANALYSIS OF FLOUR USED

	Moisture %	Ash %	Protein %	Initial pH*	Buffer pH†	Bleach
As received	7.0	0.43	11.26	5.71	3.92	Nitrogen peroxide and Novadel
to 13.5% moisture basis	....	0.40	10.48	....	....	

* Determination was made on extract prepared by adding 100 cc. of distilled water to 10 grams of the flour. Extracted one hour at 25° C. with occasional shaking. Bailey (1920) electrode, saturated calomel cell, type K Leeds and Northrup potentiometer, were used.

† Determination made on same extraction with the addition of N/50 lactic acid in the proportion of 2.5 cc. of acid to 25 cc. of the centrifuged extract.

The selection of the method of baking followed a considerable experience on different types of formulas and styles and sizes of pans. A modification of the Werner (1925) method, which has been used in this laboratory for four years in routine baking, was finally adopted because of its accuracy and convenience. The basic formula employed is:

Flour	Sugar	Salt	Yeast	Water
100 grams	2.5 grams	1.0 grams	3.0 grams	Distilled

Small doughs were mixed in jars by means of a suitable knife and large doughs in a Hobart mixer and scaled. The degree of mixing was carefully controlled, and was held uniform in all the series unless otherwise noted.

All loaves were baked in triplicate, while, for convenience, only duplicate loaves are tabulated and shown in the illustrations. All the ingredients except the flour were weighed on an analytical balance, and were dissolved in water and rinsed into the mixing bowl with the remaining water. The yeast for each series was obtained from a one-pound cake, which had first been crumbled and blended. The crease of the moulded dough was maintained in the same relative position in the pan for the entire series, with one exception, which is indicated.

All tests, unless differently shown, were handled as follows: first punch 1 hour and 35 minutes, second punch 1 hour followed by a 20-minute round-up, and a 1-hour proof. In all instances the temperature from mixer during fermentation and proofing was held at 28° C. The absorption was varied slightly because of climatic conditions. An effort was made so to control all factors and variables that results would be comparable, in that but one phase was varied for each series; while flour, procedure, method, and temperatures were constant throughout the series.

Loaf volume was measured in an accurately calibrated seed displacement apparatus, using sized mustard seed. All measurements and weights were made at specific time intervals.

Two types of pans (Fig. 1) were used, designated in the tabulations as low and tall. The low pan measured at the top 4½ x 2¾ inches, and at the bottom 3 11/16 x 2⅛ inches, with a depth of 2 inches, inside dimensions. The tall pan measured at the top 4¼ x 2 5/16 inches, and at the bottom 3 13/16 x 2 inches, with a depth of 2½ inches at the ends and 3½ inches at the sides, inside dimensions.

Two types of jars (Fig. 2) were used for the fermentation of the doughs, designated in the tabulations as low and tall. The low jar measured 5¼ inches in diameter and 2 inches in depth, inside dimensions; while the tall jar measured 4 inches in diameter and 4¾ inches in depth.

Fig. 1. Low and Tall Pans Used

Fig. 2. Low and Tall Jars Used

Different sizes and shapes of containers are used in various laboratories for the fermentation of experimental doughs. That this factor introduces a variable is indicated in Table II.

TABLE II

EFFECT OF USING DIFFERENT TYPES OF JARS FOR FERMENTING DOUGHS

Index*	Absorption %	Volume of loaf cc.	Color of crumb†	Color of crust†	Grain†	How mixed	pH on Bread‡
1	79.5	450	G	G	E el	Hand	5.5
2	79.5	455	G	G	E el	"	5.5
5	79.5	455	G+	G	E el	"	5.5
6	79.5	450	G+	G	E el	"	5.5

* The index further represents the system of indexing the loaves indicated in the illustrations.

†Key to grading: G — = good minus. G = good. G + = good plus.
E = excellent. Sl = slightly, Ti = tight.
el = elongated. sp = spherical. du = dull.

‡ Determined on the crumb of the loaf 18 hours out of the oven.

1 and 2 are duplicates fermented in tall jars and baked in tall pans, while 5 and 6 are duplicates fermented in low jars and baked in tall pans.

The grain was indistinguishable on the two series, with the character decidedly elongated and tight, while the crumb color was slightly brighter and whiter on the doughs fermented in the low jars.

The type of jar in which the doughs were fermented did not affect the loaf volume, the character of the grain, or external characteristics, but did affect the crumb color, owing to a greater expanse of the dough being exposed to the air during fermentation.

A greater loss in fermentation was also evident in doughs fermented in low jars.

In addition to the variable introduced by different styles of containers for fermenting doughs, the type of pans employed has a tremendous effect, especially on loaf volume and grain. This is shown in combination with both types of fermenting containers in Table III and illustrated by Figure 3.

TABLE III

EFFECT OF DIFFERENT STYLES OF PANS IN COMBINATION WITH DIFFERENT TYPES OF FERMENTING JARS

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	How mixed	pH on bread
1	79.5	455	G	G	E el	Machine	5.42
2	79.5	450	G	G	E el	"	
3	79.5	525	G –	G	E sp	"	5.42
4	79.5	525	G –	G	E sp	"	
5	79.5	455	G	G	E el	"	5.42
6	79.5	455	G	G	E el	"	
7	79.5	525	G –	G	E sp	"	5.42
8	79.5	520	G –	G	E sp	"	

1 and 2 are duplicates fermented in tall crocks and baked in tall pans; 3 and four are duplicates fermented in tall crocks and baked in low pans; 5 and 6 are duplicates fermented in low jars and baked in tall pans; while 7 and 8 are duplicates fermented in low jars and baked in low pans.

Fig. 3. Loaves 1 and 2 Fermented in Tall Jars and Baked in Tall Pans
Loaves 3 and 4 Fermented in Tall Jars and Baked in Low Pans
Loaves 5 and 6 Fermented in Low Jars and Baked in Tall Pans
Loaves 7 and 8 Fermented in Low Jars and Baked in Low Pans

The loaf volume was materially influenced by the type of pan used, the low pan giving a 15.4 per cent larger loaf than the tall pan, regardless of the style of fermenting jar employed. The character of the grain was also decidedly influenced, the low pan producing a spherical type of grain structure, while the tall pan produced a very elongated type. Crumb color was slightly affected by the style of pan used, the low pan producing a light dull gray cast in comparison with the loaves baked in tall pans. This may be due to the character of the grain structure, altho the tightness of the grain on both types of loaves was practically the same.

As in the first series, the type of jars had no influence on loaf volume or grain, and in this series covers both styles of pans.

It will be seen that the loaves represented by 1 and 2, of Table III and Figures 3 and 4; and those represented by 1 and 2 of Table III and Figures 5 and 6 have similar characteristics, altho the doughs of the first series were individually mixed by hand and those of the second series were mixed by machine and scaled. Both were thoroly mixed altho not for a long time in either case. Experience had completely established this factor, and in remaining tests mixing was done by machine for the same period of time with but one exception, which is indicated.

Fig. 4 Loaves 1 and 2 Handled on Normal Procedure
Loaves 3 and 4 No Punches, With Vigorous Treatment at Round-up
Loaves 5 and 6 Normal Punches, With Doughs Lightly Folded at Punch
Loaves 7 and 8 Normal Punches, With Vigorous Treatment at Punch
Loaves 9 and 10 Normal Handling With Punches Reversed

Distilled water is used in many laboratories for the experimental baking test, in order to avoid fluctuations in the character of local tap supply and in an effort to reduce a possible source of variation when collaborating on baking tests.

A duplication of the series represented by Table III was made in order to establish the possible variable introduced by using our tap water in place of distilled water. This series is represented in Table IV.

TABLE IV

EFFECT OF DIFFERENT STYLES OF PANS IN COMBINATION WITH DIFFERENT TYPES OF FERMENTING JARS, USING TAP WATER

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH on bread
9	79.5	455	G	G	E el	5.46
10	79.5	455	G	G	E el	
11	79.5	525	G –	G	E sp	5.46
12	79.5	520	G –	G	E sp	
13	79.5	455	G	G	E el	5.46
14	79.5	455	G	G	E el	
15	79.5	525	G –	G	E sp	5.48
16	79.5	520	G –	G	E sp	

9 and 10 are duplicates fermented in tall jars and baked in tall pans; 11 and 12 are duplicates fermented in tall jars and baked in

low pans; 13 and 14 are duplicates fermented in low jars and baked in tall pans; while 15 and 16 are duplicates fermented in low jars and baked in low pans.

Identical characteristics covering all phases evident in the previous series, using distilled water, were obtained in this series, using local tap water. Experience had also completely established this factor and distilled water was consequently used in the remaining tests.

The effect of tap water, however, is very pronounced in some localities, and must be considered in establishing standarized factors in experimental baking.

In order to obtain concordant results in the experimental baking test, it is quite as important to maintain a uniform system of handling the doughs as to control the mechanical phases of commercial bread production.

This is indicated in the experimental baking test by variations introduced through differences in degree of severity to which experimental doughs may be handled at the round-up. While the schedule of punching time has little influence on the character of the resultant loaf, also indicated by Harrel (1926), in comparison to other factors of dough handling, we have consistently obtained a slightly larger loaf volume through the reversal of the first and second punches in comparison with the normal system.[1] The effect of varying the severity of treatment at the round-up in comparison with a normally handled dough and a dough in which the punches have been reversed, is shown in Table V and Figure 4.

TABLE V

EFFECT OF VARYING THE HANDLING OF A DOUGH AT ROUND-UP AND OF THE REVERSAL OF NORMAL PUNCH SCHEDULE

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH on bread
1	79	450	G	G	E el	5.48
2	79	455	G	G	E el	
3	79	450	G	G	E el –	5.48
4	79	450	G	G	E el –	
5	79	470	G	G	E el	5.48
6	79	475	G	G	E el	
7	79	440	G sl +	G	E sp	5.49
8	79	435	G sl +	G	E sp	
9	79	460	G	G	E el	5.495
10	79	455	G	G	E el	

1 and 2 are duplicates handled throughout on the normal system of procedure; 3 and 4 are duplicates on which punches were

1 This fact has been known for many years.

omitted, but a very vigorous treatment was given at the round-up; 5 and 6 are duplicates on which normal punches were given, but the doughs were lightly folded at time of punching and at round-up; 7 and 8 are duplicates on which normal punches were given, but doughs were very vigorously handled; while 9 and 10 are duplicates handled on the normal system except that the first and second punches were reversed.

The external characteristics were fairly uniform on this series with a maximum spread of 35 cc. in loaf volume between 5-6 and 7-8. A slightly larger volume was obtained through the reversal of punches, while a slightly lower volume was evidenced on the normal system with severe punching. The greatest loaf volume was obtained through normal punching periods and mere folding of the doughs at punching. The series in which the punching was omitted gave a normal volume, but with slightly inferior grain characteristics. The crumb color was practically indistinguishable on the entire series with the exception of 7-8. The hydrogen-ion concentration, expressed as pH, was practically identical on the bread.

Another factor decidedly influencing the characteristics of a loaf is introduced through the mixing of dough. This has also been indicated by Harrel (1926).

Different mixers have a pronounced variable effect on the physical character of the dough (see Swanson and Working, 1926). Variances are also evidenced even when using one particular mixer at variable speeds or at different mixing periods. This is shown in Table VI and Figure 5.

TABLE VI

EFFECT OF VARIABLE MIXING ON BREAD CHARACTERISTICS

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH on bread
1	79	450	G−	G	G sl sp	5.48
2	79	450	G−	G	G sl sp	
3	79	460	G	G	G + el	5.48
4	79	460	G	G	G + el	
5	79	465	G+	G	E el	5.48
6	79	470	G+	G	E el	
7	79	480	E	G	E + el	5.48
8	79	485	E	G	E + el	

1 and 2 are duplicates mixed for 2 minutes at slow speed, 1 minute at medium speed, and 2 minutes at fast speed; 3 and 4 are duplicates mixed for 2 minutes at slow speed, and 3 minutes at fast speed; 5 and 6 are duplicates mixed for 1 minute at slow speed and 5

minutes at fast speed; 7 and 8 are duplicates mixed for 2 minutes at slow speed and 10 minutes at fast speed.

Fig. 5. Loaves 1 and 2 Mixed 2 min. at Slow Speed, 1 min. at Medium, and 2 min. at Fast Speed
Loaves 3 and 4 Mixed 2 min. at Slow Speed, 3 min. at Fast Speed
Loaves 5 and 6 Mixed 1 min. at Slow Speed, 5 min. at Fast Speed
Loaves 7 and 8 Mixed 2 min. at Slow Speed, 10 min. at Fast Speed

Loaf volume increased and grain and crumb characteristics improved with each increase in mixing time at fast speed. The loaves represented by 1-2 and 7-8 were of entirely different character. The similar hydrogen-ion concentration, expressed as pH, of the bread indicates that the mixer action was purely mechanical.

The placement of the dough crease in panning has a particular effect upon the external characteristics of the loaf. The loaf must be maintained in the same relative position to insure uniformity in test baking. This is shown in Table VII and Figure 6.

Fig. 6. Loaves 21 and 22—Dough Crease Centered on Bottom
Loaves 23 and 24—Dough Crease Centered on One Side
Loaves 25 and 26—Dough Crease Centered on Top

TABLE VII

EFFECT OF VARYING THE RELATIVE POSITION OF THE DOUGH CREASE AT PANNING

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	Break
21	79	460	G	G	E	Smooth
22	79	460	G	G	E	"
23	79	460	G	G	E–	Fairly smooth
24	79	455	G	G	E–	" "
25	79	465	G	G	E–	Irregular
26	79	465	G	G	E–	"

21 and 22 are duplicates on which the dough crease was placed in the normal way, centered on the bottom; 23 and 24 are duplicates on which the crease was centered on one side; while 25 and 26 are duplicates on which the crease was centered on top.

Very irregular shaped loaves resulted from placing the crease on top, and a slightly inferior grain was evidenced on both these and the loaves on which the crease was centered on the side.

The addition of varying amounts of dough ingredients other than those employed in a simple test formula affect the characteristics of the resultant loaf. The introduction into the formula of a non-diastatic malt is indicated in Table VIII and Figure 7.

TABLE VIII

EFFECT OF ENRICHING THE BASIC FORMULA WITH VARYING AMOUNTS OF NON-DIASTATIC MALT EXTRACT

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH on bread
15	79	450	G	G	E el	5.53
16	79	450	G	G	E el	
17	79	450	G+	G	E el	5.53
18	79	455	G+	G	E el	
19	79	455	E	E	E el	5.46
20	79	460	E	E	E el	
21	79	465	E−	*	E el	5.42
22	79	465	E−	*	E el	

15 and 16 are duplicates from the basic formula; 17 and 18 are duplicates to which ½ of 1 per cent of non-diastatic malt was added; 19 and 20 are duplicates to which 1 per cent of non-diastatic malt was added; while 21 and 22 are duplicates to which 2 per cent of non-diastatic malt was added.

Fig. 7. Loaves 15 and 16 Made from Basic Formula
Loaves 17 and 18 Same Plus 0.5% Non-diastatic Malt
Loaves 19 and 20 Same Plus 1 % Non-diastatic Malt
Loaves 21 and 22 Same Plus 2 % Non-diastatic Malt

The crumb color was slightly improved by adding ½ of 1 per cent malt extract, and decidedly improved by adding 1 per cent. The crumb was quite white, but in the doughs to which 2 per cent malt was added, was of a dull gray cast.

The grain was practically indistinguishable on the series and the loaf volume was but slightly influenced. The crust color was deepened in intensity with increasing amounts of non-diastatic malt extract, the addition of 2 per cent giving a rich dark golden brown color.

The result of adding varying amounts of diastatic malt extract of 250 degrees Lintner is more pronounced. This is shown in Table IX and Figures 8 and 9.

TABLE IX

EFFECT OF ENRICHING THE BASIC FORMULA WITH VARYING AMOUNTS OF HIGH DIASTATIC MALT EXTRACT

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH on bread
13	78	450	G	G	E el	5.48
14	78	455	G	G	E el	
15	77	530	G+	G+	E el	5.41
16	77	535	G+	G+	E el	
17	76	560	E	E	G sl sp	5.39
18	76	560	E	E	G sl sp	
19	75	590	E+	E+	G sp	5.32
20	75	595	E+	E+	G sp	

13 and 14 are duplicates from the basic formula; 15 and 16 are duplicates to which 1 per cent of high diastatic malt was added; 17 and 18 are duplicates to which 2 per cent of high diastatic malt was added; while 19 and 20 are duplicates to which 3 per cent of high diastatic malt was added.

Fig. 8. Loaves 13 and 14 Made from Basic Formula
Loaves 15 and 16 Same Plus 1% High Diastatic Malt Extract
Loaves 17 and 18 Same Plus 2% High Diastatic Malt Extract
Loaves 19 and 20 Same Plus 3% High Diastatic Malt Extract

Fig. 9. Loaf 13 Made from Basic Formula
Loaf 15 Same Plus 1% High Diastatic Malt
Loaf 17 Same Plus 2% High Diastatic Malt
Loaf 19 Same Plus 3% High Diastatic Malt

Absorption in this series was decreased 1 per cent for each 1 per cent addition of malt extract.

Loaf volume showed a decided increase and crumb color a pronounced improvement with increasing amounts of high diastatic malt extract. The grain was not injured by the addition of 1 per cent malt, but was seriously affected by the addition of 2 and 3 per cent.

Fig. 10. Loaves 24 and 25 Made from Basic Formula
Loaves 26 and 27 Same Plus 0.5% Powdered Milk
Loaves 28 and 29 Same Plus 1 % Powdered Milk
Loaves 30 and 31 Same Plus 2 % Powdered Milk

The crust was materially darkened by the addition of increasing quantities of high diastatic malt. All loaves in this series were well formed and there was no indication of slacking during fermentation, altho the bread which carried 2 per cent and 3 per cent of malt had little body. The hydrogen-ion concentration, expressed as pH, of the bread showed a decided difference and the odor of malt was pronounced on the last two sets of the series.

Adding varying amounts of milk to the basic formula affects bread characteristics. The effect of using powdered milk is indicated in Table X and in Figure 10.

TABLE X

EFFECT OF ADDING VARYING AMOUNTS OF POWDERED MILK TO THE BASIC FORMULA

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
24	79	450	G	G	E el
25	79	455	G	G	E el
26	79	450	G	G sl +	E + el
27	79	450	G	G + sl	E + el
28	79	440	G −	G +	E + sp
29	79	440	G −	G +	E + sp
30	79	435	P	E	G sp
31	79	435	P	E	G sp

24 and 25 are duplicates from the basic formula; 26 and 27 are duplicates to which ½ of 1 per cent of powdered milk was added; 28 and 29 are duplicates to which 1 per cent of powdered milk was added; 30 and 31 are duplicates to which 2 per cent of powdered milk was added. The absorption was not increased to compensate for the milk in this series.

Increasing amounts of powdered milk injured the loaf volume, crumb color, and grain characteristics, but improved the crust color. The grain was decidedly spherical on the loaves containing 1 per cent and 2 per cent of powdered milk. These two loaves were fairly tight at the round-up. A decided milk odor was evident on the series containing 1 per cent powdered milk, and very pronounced on the series containing 2 per cent.

Fig. 11. Loaves 12 and 13 Made from Basic Formula
Loaves 14 and 15 Same Plus 0.25% "A"
Loaves 16 and 17 Same Plus 0.5 % "A"
Loaves 18 and 19 Same Plus 0.75% "A"

The addition of certain mineral salts to the basic formula materially alters the bread characteristics. The effect of using varying amounts of a commercial product[2] is shown in Table XI and Figures 11 and 12.

Fig. 12. Loaf 12 Made From Basic Formula
Loaf 14 Same Plus 0.25% "A"
Loaf 16 Same Plus 0.5 % "A"
Loaf 18 Same Plus 0.75% "A"

TABLE XI

EFFECT OF ADDING VARYING AMOUNTS OF MINERAL SALTS "A" TO THE BASIC FORMULA

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH dough to oven	pH bread
12	79	455	G	G	E el	5.42	5.48
13	79	450	G	G	E el		
14	79	465	G+	G	E sl sp	5.28	5.33
15	79	465	G+	G	E sl sp		
16	79	480	E	G−	G sp	5.02	5.17
17	79	485	E	G−	G sp		
18	79	460	E+	P	G− sp	4.94	5.04
19	79	465	E+	P	G− sp		

[2] A patented mixture of ammonium chloride, calcium sulfate, and potassium bromate.

12 and 13 are duplicates from the basic formula; 14 and 15 are duplicates to which ¼ of 1 per cent of A was added; 16 and 17 are duplicates to which ½ of 1 per cent of A was added; while 18 and 19 are duplicates to which ¾ of 1 per cent of A was added. The punches were reversed on the doughs which contained A.

Loaf volume was increased and crumb color improved through the addition of ¼ of 1 per cent and ½ of 1 per cent A, while the grain character and the crust color were slightly injured. The use of ¾ of 1 per cent A produced a white crumb with a dull cast, with inferior grain structure and decidedly pale crust.

The ageing effect of A is indicated by the increase in hydrogen-ion concentration, expressed as pH, on the doughs (time to oven) and the bread.

The effect of using varying amounts of B[3] is indicated in Table XII and Figure 13.

Fig. 13. Loaves 13 and 14 Made from Basic Formula
Loaves 15 and 16 Same Plus 0.25% "B"
Loaves 17 and 18 Same Plus 0.5 % "B"
Loaves 19 and 20 Same Plus 0.75% "B"

TABLE XII

EFFECT OF ADDING VARYING AMOUNTS OF B TO THE BASIC FORMULA

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
13	79	450	G	G	E el
14	79	450	G	G	E el
15	79	455	G+	G	E + el
16	79	450	G+	G	E + el
17	79	455	E	G−	E sl sp
18	79	460	E	G−	E sl sp
19	79	405	G−	G−	G − sp
20	79	400	G−	G−	G − sp

13 and 14 are duplicates from the basic formula; 15 and 16 are duplicates to which ¼ of 1 per cent of B was added; 17 and 18 are duplicates to which ½ of 1 per cent of B was added; 19 and 20 are duplicates to which ¾ of 1 per cent of B was added.

[3]B is a mixture of calcium peroxide and inorganic phosphates, extensively used as a dough developer.

The doughs became progressively tighter with increasing amounts of B.

The crumb color was decidedly improved and the loaf volume slightly increased by the addition of ¼ of 1 per cent and ½ of 1 per cent B, while the grain character was improved by the addition of ¼ of 1 per cent.

The addition of ¾ of 1 per cent B materially injured the loaf volume, crumb color, grain structure, and crust color.

One of the ingredients frequently employed in experimental baking is a shortening agent. The effect of adding varying amounts of C[4] to the dough batch is shown in Table XIII and Figure 14.

Fig. 14. Loaves 13 and 14 Made from Basic Formula
Loaves 15 and 16 Same Plus 1% "C"
Loaves 17 and 18 Same Plus 2% "C"
Loaves 19 and 20 Same Plus 3% "C"

TABLE XIII

EFFECT OF ADDING VARYING AMOUNTS OF C TO THE BASIC FORMULA

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH on bread
13	79	455	G	G	E el	5.48
14	79	455	G	G	E el	
15	79	455	G+	G	E+ el	5.46
16	79	460	G+	G	E+ el	
17	79	460	E	G+	E+ v el	5.46
18	79	455	E	G+	E+ v el	
19	79	420	E sl+	G+	E+ v ti	5.46
20	79	425	E sl+	G+	E+ v ti	

13 and 14 are duplicates from the basic formula; 15 and 16 are duplicates to which 1 per cent of C was added; 17 and 18 are duplicates to which 2 per cent of C was added; 19 and 20 are duplicates to which 3 per cent of C was added.

Crumb color, grain structure, and crust color were improved with increasing amounts of C. Loaf volume was not materially

[4] C is represented as a hydrostearoleine.

affected by the addition of 1 and 2 per cent of C but was decidedly decreased by the addition of 3 per cent.

Some form of sugar is used in all baking tests, altho the percentage varies. The effect of using various amounts of cane sugar is shown in Table XIV and in Figure 15.

Fig. 15. Loaf 12 With 1.5% Sucrose
Loaf 14 With 2 % Sucrose
Loaf 16 With 2.5% Sucrose
Loaf 18 With 3.5% Sucrose

TABLE XIV

EFFECT OF USING VARYING AMOUNTS OF CANE SUGAR

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
12	79	445	G −	G −	E sl sp
13	79	450	G −	G −	E sl sp
14	79	450	G	G	E el
15	79	450	G	G	E el
16	79	455	G +	G +	E + el
17	79	460	G +	G +	E + el
18	79	465	E sl du	E	E + sp
19	79	465	E sl du	E	E + sp

12 and 13 are duplicates using 1½ per cent of sugar; 14 and 15 are duplicates using 2 per cent of sugar; 16 and 17 are duplicates using 2½ per cent of sugar; 18 and 19 are duplicates using 3½ per cent of sugar.

The crumb and crust color and the grain structure were improved, and the volume slightly increased with increasing amounts of cane sugar up to 2½ per cent. The loaves containing 3½ per cent sugar, altho the whitest of the series, had a slightly dull cast on the crumb and the grain structure was decidedly spherical but very tight in character.

The effect of substituting Cerelose for cane sugar in varying amounts is indicated in Table XV and Figure 16.

TABLE XV

EFFECT OF SUBSTITUTING CERELOSE FOR CANE SUGAR IN VARYING AMOUNTS

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
14	79	450	G	G	E el
15	79	455	G	G	E el
16	79	440	G –	G –	G sl sp
17	79	440	G –	G –	G sl sp
18	79	455	G	G	E el
19	79	450	G	G	E el
20	79	445	E sl du	G +	E + sl sp
21	79	440	E sl du	G +	E + sl sp

14 and 15 are duplicates from the basic formula; 16 and 17 are duplicates using 1 per cent Cerelose; 18 and 19 are duplicates using 2 per cent Cerelose; 20 and 21 are duplicates using 3 per cent Cerelose.

The crumb and crust color and grain structure were improved and the loaf volume slightly increased with increasing amounts of Cerelose up to 2 per cent. The doughs containing 3 per cent Cerelose were slightly sticky, and altho producing the whitest loaf, had a slightly dull crumb. The grain structure was slightly spherical but very tight.

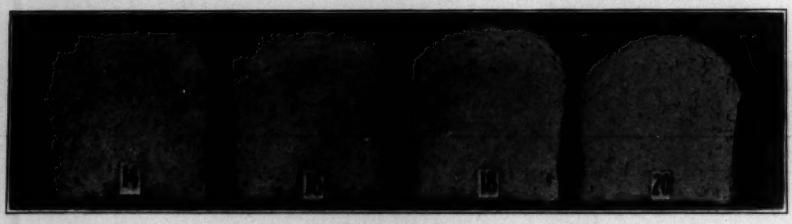

Fig. 16. Loaf 14 Made from Basic Formula
Loaf 16 Same With 1% Cerelose
Loaf 18 Same With 2% Cerelose
Loaf 20 Same With 3% Cerelose

Doughs containing 2 per cent Cerelose produced loaves indistinguishable from those on the basic formula.

The quantity of yeast employed in experimental test doughs is quite variable. The result of using different percentages in the dough is shown in Table XVI.

The loaf volume increased with increasing amounts of yeast, with a spread of 40 cc. between the doughs containing 1½ per cent and 4 per cent. The crumb and crust color likewise improved up to 3 per cent of yeast while the loaves from the doughs containing 4 per cent of yeast showed considerable age. The grain structure was affected by the use of increased amounts of yeast.

TABLE XVI
EFFECT OF USING VARYING AMOUNTS OF YEAST

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
3	79	430	G –	G –	E + el
4	79	435	G –	G –	E + el
5	79	440	G –	G	E el
6	79	440	G –	G	E el
7	79	450	G	G	E el
8	79	455	G	G	E el
9	79	465	G – du	G –	E sp
10	79	470	G – du	G –	E sp

3 and 4 are duplicates using 1½ per cent of yeast; 5 and 6 are duplicates using 2 per cent of yeast; 7 and 8 are duplicates from the basic formula; 9 and 10 are duplicates using 4 per cent of yeast.

Using fresh yeast has been consistently advocated for commercial bread production as well as for experimental baking tests. In an effort to establish this factor, we have obtained some very startling results through using yeast stored at 65° F. in the original wrapper for varying lengths of time. The yeast, in all cases regardless of length of storage, gave no indication of spoilage. This series has been rechecked only four times, yet the results have been quite concordant, as represented in Tables XVII and XVIII. Final results are illustrated in Figure 17.

Fig. 17. Loaves 14 and 15 Made With Fresh Yeast
Loaves 16 and 17 Made With 1 Day Old Yeast
Loaves 18 and 19 Made With 2 Day Old Yeast
Loaves 20 and 21 Made With 3 Day Old Yeast

TABLE XVII
EFFECT OF USING YEAST OF DIFFERENT DEGREES OF FRESHNESS

Index	Yeast age	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH on bread
14	Fresh	79	455	G	G	E el	5.48
15	Fresh	79	450	G	G	E el	
16	1 day	79	455	G sl –	G	E el	5.49
17	1 day	79	455	G sl –	G	E el	
18	2 days	79	445	G	G	E + el	5.49
19	2 days	79	445	G	G	E + el	
20	3 days	79	475	G	G	E sl + el	5.48
21	3 days	79	475	G	G	E sl + el	

The variation was very slight on the entire series except for the increased loaf volume on the set with yeast 3 days old.

TABLE XVIII

EFFECT OF USING YEAST OF DIFFERENT DEGREES OF FRESHNESS

Index	Yeast age	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
20	Fresh	79	450	G	G	E el
21	Fresh	79	450	G	G	E el
22	1 day	79	450	G	G	E el
23	1 day	79	445	G	G	E el
24	2 days	79	470	G+	G	E sl+ el
25	2 days	79	470	G+	G	E sl+ el
26	3 days	79	455	G sl +	G	E sl + el
27	3 days	79	460	G sl +	G	E sl + el

The variation on this series, again, was very slight, except for increased loaf volume and improved crumb color on the set with yeast 2 days old.

Fig. 18. Loaf 26 Made With Yeast "D"
Loaf 28 Made With Yeast "E"

In a comparison of the two principal brands of yeast available, "D" and "E", for the experimental baking test, variations in dough and bread characteristics were observed which, however, did not remain constant on all factors in successive tests. A comparison is shown in Table XIX and Figures 18, 19, 20, and 21.

Fig. 19. Loaves 27 and 28 Made With Yeast "D"
Loaves 29 and 30 Made With Yeast "E"

TABLE XIX

COMPARATIVE BAKING TESTS USING D AND E YEAST

Index	Yeast	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH on bread
26	D	79	460	G	G	E el	5.47
27	D	79	455	G	G	E el	
28	E	79	455	G+	G	E el+ sl	5.369
29	E	79	455	G+	G	E el+ sl	
27	D	79	455	G	G	E el	5.48
28	D	79	455	G	G	E el	
29	E	79	465	G sl+	G	E el	5.38
30	E	79	460	G sl+	G	E el	
20	D	79	465	G	G	E el	5.44
21	D	79	470	G	G	E el	
22	E	79	460	G	G	E el	5.40
23	E	79	455	G	G	E el	
13	D	79	465	G	G	E el	5.46
14	D	79	465	G	G	E el	
15	E	79	475	G sl+	G	E sl+ el	5.35
16	E	79	470	G sl+	G	E sl+ el	

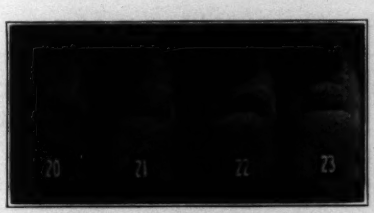

Fig. 20. Loaves 20 and 21 Made With Yeast "D"
Loaves 22 and 23 Made With Yeast "E"

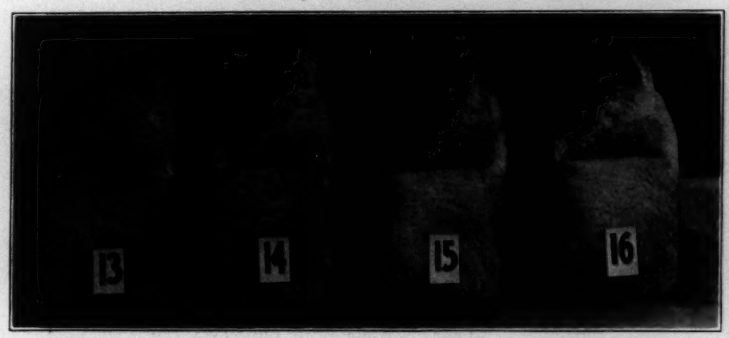

Fig. 21. Loaves 13 and 14 Made With Yeast "D"
Loaves 15 and 16 Made With Yeast "E"

Loaves baked from doughs containing E yeast consistently carried a slightly higher hydrogen-ion concentration, expressed as pH, than loaves baked from doughs containing D yeast. This undoubtedly explains the tendency toward slightly better crumb color

and grain structure evident in some instances on bread from E yeast doughs. The loaf volume varied 20 cc. on the entire series, and was practically comparable on individual comparative bakes.

The height of the E doughs was consistently two-thirds the height of the D doughs at the time of first punch, while the height in the pan following the proofing was practically identical.

The amount of salt used in experimental test doughs affects the bread characteristics. This is shown in Table XX and Figure 22.

TABLE XX
EFFECT OF USING VARYING AMOUNTS OF SALT

Index	Amount of salt %	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
13	½	79	460	G −	G −	G + el
14	½	79	460	G −	G −	G + el
15	1	79	455	G	G	E el
16	1	79	450	G	G	E el
17	1½	79	440	G+	G	E sp ti
18	1½	79	435	G+	G	E sp ti
19	2½	79	415	G −	G −	E + v ti
20	2½	79	415	G −	G −	E + v ti

The loaf volume decreased with increasing amounts of salt, with a spread of 45 cc. between the doughs containing ½ of 1 per cent and 2½ per cent. The crumb improved up to 1½ per cent of salt while the doughs containing 2½ per cent were decidedly young. The grain improved with increasing amounts of salt, changing in character, however, from an elongated cell structure on the ½ of 1 per cent salt series, to a very tight spherical structure on the 2½ per cent series.

Fig. 22. Loaf 13 Made With 0.5% Salt
Loaf 15 Made With 1.0% Salt
Loaf 17 Made With 1.5% Salt
Loaf 19 Made With 2.5% Salt

The deleterious effect of excessive pan greasing on the volume of loaves baked in commercial type bread pans has been shown by Harrel (1926).

This influence is likewise maintained on loaves baked in narrow test type bread pans as indicated in Table XXI and Figure 23.

TABLE XXI

EFFECT OF VARYING AMOUNTS OF PAN GREASE

Index	How greased	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
25	None	79	450	G	G	E el
26	None	79	450	G	G	E el
27	Light	79	440	G	G	E el
28	Light	79	445	G	G	E el
29	Heavy	79	430	G + sl	G	E sl + el
30	Heavy	79	430	G + sl	G	E el + el

The loaf volume was materially injured by increasing the pan greasing.

Fig. 23. Loaf 25 No Grease in Pan
Loaf 27 Light Grease in Pan
Loaf 29 Heavy Grease in Pan

It is difficult before baking to obtain the exact absorption best suited for a flour. The effect on the bread characteristics of varying the absorption is shown in Table XXII and Figure 24.

Fig. 24. Loaves 19 and 20 With 79% Absorption
Loaves 21 and 22 With 80% Absorption
Loaves 23 and 24 With 78% Absorption
Loaves 25 and 26 With 80% Absorption

TABLE XXII

EFFECT OF VARYING THE ABSORPTION

Index	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
19	79	450	G	G	E el
20	79	455	G	G	E el
21	80	440	G sl −	G	E sl − el
22	80	445	G sl −	G	E sl − el
23	78	460	G sl +	G	E sl + el
24	78	460	G sl +	G	E sl + el
25	80*	465	G +	G	E + el ti
26	80	460	G +	G	E + el ti

* This series was mixed for 2 minutes at slow speed and 15 minutes at high speed.

A variation in absorption of only 1 per cent above and below normal, slightly affected loaf volume and crumb color.

The series indexed as 25-26 carried 2 per cent more absorption than the series indexed as 23-24 and yet produced bread of comparable volume with improved crumb color and grain character. This indicates the necessity of balancing the absorption with mixing procedure if optimum and concordant results are desired.

Length of fermentation period is considered of extreme importance in relation to the proper developing of the dough in order to obtain optimum baking results.

This phase of baking is decidedly critical with some flours while other types of flour possess a relatively wide margin in which satisfactory bread can be produced. Harrel (1926) has shown the possible variation in this spread on the same flour when used on commercial straight and sponge doughs. The effect of improper time of fermentation is generally more pronounced in commercial practice than in experimental baking, owing to the amount of dough, the type of formula, and the severity of machine treatment.

The effect of varying the fermentation period on the basic flour of this series is indicated in Table XXIII and Figure 25.

Fig. 25. Loaves 24 and 25 With 2:25 hr. Trough Fermentation
Loaves 26 and 27 With 2:55 hr. Trough Fermentation
Loaves 28 and 29 With 3:25 hr. Trough Fermentation
Loaves 30 and 31 With 3:55 hr. Trough Fermentation

TABLE XXIII

EFFECT OF VARYING THE FERMENTATION PERIOD

Index	Fermentation period	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
24	2 25	79	450	G sl −	G	E sl + el
25	2 25	79	450	G sl −	G	E sl + el
26	2 55	79	455	G	G	E el
27	2 55	79	450	G	G	E el
28	3 25	79	460	G sl +	G	E el
29	3 25	79	460	G sl +	G	E el
30	3 55	79	465	G −	G −	E sp
31	3 55	79	470	G −	G −	E sp

With this flour a spread of 1 hour and 30 minutes in fermentation time varied the loaf volume only 20 cc. and indicated slight variations in grain structure, crumb color, and crust color.

Fig. 26. Loaf 17 With 2:55 hr. Trough Fermentation
Loaf 19 With 2:30 hr. Trough Fermentation
Loaf 21 With 2:00 hr. Trough Fermentation
Loaf 23 With 1:30 hr. Trough Fermentation

The effect of varying the fermentation period on a flour of limited margin is decidedly more pronounced. A 95 per cent straight grade flour[5] milled from very ordinary wheat of low protein content and handled on varying fermentation periods is indicated in Table XXIV and Figure 26.

TABLE XXIV

EFFECT OF VARYING THE FERMENTATION PERIOD ON A FLOUR OF LIMITED MARGIN

Index	Fermentation period	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH on bread
17*	2 55	79.0	450	G	G	E el	5.48
18	2 55	79.0	455	G	G	E el	
19	2 30	61.5	420	G −	G −	E sl sp	5.325
20	2 30	61.5	425	G −	G −	E sl sp	
21	2 00	61.5	435	G sl −	G	G + el	5.36
22	2 00	61.5	440	G sl −	G	G + el	
23	1 30	61.5	490	G	G	G sl el	5.42
24	1 30	61.5	495	G	G	G sl el	

*Basic dough.

[5]Moisture 13.9%, ash 0.43%, protein 10.2%, initial hydrogen-ion concentration expressed as pH 5.67, buffer 3.89.

With this flour a spread of 1 hour in fermentation time varied the loaf volume 75 cc., and variations in the grain structure and crumb color were rather pronounced. The loaf volume of this flour on 1 hour and 30 minutes fermentation was considerably larger than the basic series, while the grain structure was inferior.

A pronounced effect is obtained on the bread characteristics by varying the proofing period. This is indicated in Table XXV and in Figure 27.

Fig. 27. Loaves 21 and 22 With 1:20 hr. Proofing
Loaves 23 and 24 With 1:10 hr. Proofing
Loaves 25 and 26 With 1:00 hr. Proofing
Loaves 27 and 28 With 0:45 hr. Proofing

TABLE XXV

EFFECT OF VARYING THE PROOFING PERIOD

Index	Proof period	Absorption %	Volume of loaf	Color of crumb	Color of crust	Grain
			cc.			
21	1 20	79	480	G −	G	G sl sp
22	1 20	79	480	G −	G	G sl sp
23	1 10	79	470	G −	G	G + sl sp
24	1 10	79	465	G −	G	G + sl sp
25	1 00	79	450	G	G	E el
26	1 00	79	455	G	G	E el
27	0 45	79	435	G+	G sl −	E + el
28	0 45	79	430	G+	G sl −	E + el

The loaf volume was decidedly increased while the grain structure and crumb color were injured by increased periods of pan proofing.

It has been shown that bread characteristics are modified by variations in dough formula as well as by mechanical procedure. The type of dough employed also influences the character of the loaf.

Loaves baked from commercial type straight doughs[6] with varying fermentation periods and from commercial type sponge doughs[7]

[6] Formula: Flour 392 gm., salt 7 gm., sugar 8 gm., powdered milk 6 gm., malt extract 4 gm., yeast 7 gm., commercial bread improver 1 gm., and all hydrogenated shortening 8 gm.

[7] Formula: Sponge, flour 240 gm., yeast 8 gm., and commercial bread improver 2 gm. Dough, flour 160 gm., sugar 8 gm., powdered milk 6 gm., salt 7 gm., all hydrogenated shortening 7 gm., and malt extract 4 gm.

show considerable differences. This is indicated in Table XXVI and in Figure 28.

TABLE XXVI

COMMERCIAL TYPE STRAIGHT DOUGHS ON VARYING FERMENTATION PERIODS IN COMPARISON WITH A COMMERCIAL TYPE SPONGE DOUGH

Index	Type of dough	Fermentation period	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain	pH on bread
18	Straight	2 30	79	480	G	G	E sl sp	5.51
19	Straight	2 30	79	475	G	G	E sl sp	
20	Straight	3 00	79	485	G+	G sl+	E sl+ el	5.42
21	Straight	3 00	79	490	G+	G sl+	E sl+ el	
22	Straight	3 30	79	495	G+sl du	G sl−	E el	5.34
23	Straight	3 30	79	500	G+sl du	G sl−	E el	
24	Sponge	4 53	79	555	E	G sl+	E+ el	5.29
25	Sponge	4 53	79	550	E	G sl+	E+ el	

Fig. 28. Loaves 18 and 19 Commercial Type Straight Dough With 2:30 hr. Fermentation
Loaves 20 and 21 Commercial Type Straight Dough With 3:00 hr. Fermentation
Loaves 22 and 23 Commercial Type Straight Dough With 3:30 hr. Fermentation
Loaves 24 and 25 Commercial Type Sponge Dough With 4:53 hr. Fermentation

With the commercial type straight dough a spread of 1 hour in fermentation time varied the loaf volume 25 cc. and indicated rather pronounced variations in grain structure, crumb and crust color. The hydrogen-ion concentration expressed as pH on these loaves varied over a spread of 0.17.

Fig. 29. Loaves 26 and 27 Molded Medium Tight
Loaves 28 and 29 Molded Loose
Loaves 30 and 31 Molded Very Tight

A considerably larger loaf was obtained with the commercial type sponge dough, while the crumb color and grain structure were slightly improved. These loaves had decidedly less body and slightly inferior flavor.

In the experimental baking test the technic of certain phases might very easily influence the interpretation of results. The effect of molding the dough to different degrees of tightness is shown in Table XXVII and Figure 29.

TABLE XXVII

EFFECT OF MOLDING TO DIFFERENT DEGREES OF TIGHTNESS

Index	How molded	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
26	Medium	79	450	G	G	E el
27	Medium	79	455	G	G	E el
28	Loose	79	460	G sl −	G	G + sl sp
29	Loose	79	460	G sl −	G	G + sl sp
30	Tight	79	445	G sl +	G	E + el
31	Tight	79	440	G sl +	G	E + el

The loaf volume was slightly decreased and the grain and crumb color improved through tight molding of the dough, while loose molding slightly injured the grain and crumb color and improved the loaf volume.

The effect of different oven temperatures is shown in Table XXVIII and Figure 30.

Fig. 30. Loaf 14 Baked at 325° F.
Loaf 16 Baked at 375° F.
Loaf 18 Baked at 425° F.

TABLE XXVIII

EFFECT OF DIFFERENT OVEN TEMPERATURES

Index	Oven temperature °F.	Absorption %	Volume of loaf cc.	Color of crumb	Color of crust	Grain
14	325	79	480	G du	G −	E + el
15	325	79	475	G du	G −	E − el
16	375	79	460	G	G	E el
17	375	79	460	G	G	E el
18	425	79	450	G	G +	E el
19	425	79	455	G	G +	E el

An oven temperature of 325° F. produced the largest loaf, the loaves, however, having a very thick crust and slightly inferior crumb and crust color and grain structure.

Loaf volume was decreased with increasing oven temperatures, with a slight improvement in grain structure and crumb color.

Summary

The experimental baking test is the most valuable method for determining flour characteristics.

The interpretation of results as well as the uniformity of results is readily influenced by variations in technic, method of procedure, apparatus employed, mechanical treatment, and formula.

These influences are indicated in part by the effect of size and shape of fermenting jars and baking pans, type of water, punching schedule, severity of punching, different mixing time and speed, placement of dough crease, use of varying amounts of non-diastatic and high-diastatic malt extracts, powdered milk, mineral salts, shortening, cane sugar, Cerelose, yeast, salt, and pan greasing, the degree of freshness of the yeast, the type of yeast, the fermentation period, the proofing period, and oven temperatures.

Acknowledgments

Grateful acknowledgment is made of the indebtedness of the senior author to E. E. Werner for his communication of the foundations on which this type of test baking is based, and for his assistance on many occasions; and to William Meyers and Max Markley for the analytical data.

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A STUDY OF THE RATE OF DRYING OF WHEAT FLOUR, STARCH, AND GLUTEN

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Introductory

The moisture content of wheat and flour is a characteristic of great importance. Not only are wheat and flour bought and sold on a weight basis, but the water content of the material has a very intimate connection with the keeping quality. Moreover, the water content of wheat and flour is extremely variable: during the cereal years 1919-21 the U. S. Department of Agriculture, Bureau of Agricultural Economics, examined 103,680 samples of wheat from all parts of the country, and found the moisture contents to vary from 5.0 to 24.7 per cent. The moisture content of flour is also very variable, although fewer data are to be found in the literature. The reason of this great variation is to be found in the *hygroscopicity* of the material; that is, the power of the material to absorb moisture from, or give up moisture to, the surrounding atmosphere according to the temperature and humidity of the latter. When flour is handled, stored, or transported in ordinary commercial practice it may absorb moisture and increase in weight or it may partially dry and lose weight, according to the temperature and humidity conditions of the surrounding atmosphere. C. H. Bailey (1920) studied the effect of atmospheric humidity on moisture content of flour and showed that moisture content may vary from 5% to 15% as the relative humidity of the air increases from 30 to 80%. Some of his results are given in Table I.

TABLE I

Hygroscopic Moisture of Patent and Second Clear Flours in Contact with Atmospheres of Various Humidities (Bailey, 1920)

Relative humidity of atmosphere at 25°C. (= 78°F.)	Moisture content of flour	
	Patent	Second
%	%	%
29.4	5.18	5.11
50.3	7.92	7.81
69.8	12.05	11.65
80.0	15.00	15.00

Experiments of Stockham (1917) indicated that flour kept for some time in a still atmosphere saturated with water vapour might take up as much as 34.8% of moisture.

The moisture content of wheat and flour is so variable and at the same time so important that it is not surprising that a great deal of attention has been given to its determination. No work, however, appears to have been carried out on the rate at which wheat and flour will absorb moisture from or give up moisture to the atmosphere in response to varying humidity conditions. Extensive investigations on various moisture relations of wheat and flour are in progress in the writer's laboratories. This communication is concerned only with the rate of drying of flour when the flour is exposed at constant temperature to atmospheres of approximately zero humidity.

Experimental

The experimental method consisted in exposing flour over concentrated sulphuric acid in a closed vessel at a constant temperature; and the apparatus was so arranged that the flour could be weighed at intervals without removing it from the drying vessel or in any way disturbing the drying process or the drying conditions.

The apparatus was essentially that devised by Keen (1914) for studying the evaporation of water from soil. As it is described and illustrated in his original paper, a short description only is necessary here. This apparatus is now on the market and is sold by A. Gallenkamp & Co. It was used by Fisher (1923a, 1923b, 1924a) in his earlier work on the drying of wool, soil, gelatin, and other colloidal materials. The containers used by the writer were circular aluminum pans 2¾ inches in diameter and about ½ inch deep, hung from stout copper wires which had hooks at the upper ends. The container stood on a glass tripod in a glass drying vessel 6 × 3 × 3 inches, in the bottom of which was 50 to 60 cc. of concentrated sulphuric acid. The upper edges of the drying vessel were ground flat, and glass cover plates were cemented on with vaseline. The cover plate had a circular hole 0.5 cm. in diameter bored through the center, and the plate was cut in two transversely across this center hole. This allowed the two half-cover plates to be fitted in position after the container with flour had been put in the vessel, the copper wire stem passing through the center hole of the cover plate. Two similar but smaller half-cover plates were placed above the lower ones; the center hole in these was just big

enough to fit closely round the copper wire support of the containers. All edges in contact were coated with vaseline. The drying vessels (six) were fitted radially into a circular zinc thermostat to within about one-eighth inch of the cover plate. The thermostat was mounted on a circular rail which rested on wheels attached to the base plate of the apparatus so that it could be rotated to bring each container in succession under a balance which was supported by a table. A vertical copper wire attached to the under side of the left balance pan passed downward through holes in the base of the balance and table and terminated in a small hook, into which the hook of the copper wire support of each container could be fastened and the container weighed *in situ*. During each weighing the two upper small half-cover plates were moved apart, thus exposing the larger hole in the lower cover plates through which the metal container could swing freely while being weighed. In this way the percentage moisture content (calculated on a dry weight basis) could be determined for each time the material was weighed, and when these were plotted against the time (expressed in minutes from the commencement of the drying operation) the points obtained were found to lie on a smooth curve.

Some Typical Drying Curves

The data obtained with a sample of a patent blended flour, (City Imperial from New Barnes Mill), are given in Table II.[1]

The percentage moisture contents are plotted against time of drying in Figure I (Curve A). This curve, which may be called the experimental curve, obviously represents the variation in percentage moisture content (calculated on a dry weight basis) with time of drying. By plotting this curve on a large scale and measuring the tangents by means of a straight edge at different points along it, values were obtained for the instantaneous rates of drying at different water contents under the particular constant drying conditions employed. These rates of drying plotted against

1 The drying was continued for 39 days, the lowest weight recorded being taken as the dry weight. After two weeks drying the apparent dry weight fluctuated slightly from day to day. The last traces of moisture are held by flour with great tenacity and it has been shown by the writer [Res. Ass. British Flour Millers Confidential Report No. 1 (1925); see also O. Nelson and Hulett (1920)], that a true determination of moisture content cannot be made by any method of oven drying. Moreover, it was not found practicable to seal hermetically the drying vessel, so that a small leakage of outside air into the apparatus may have occurred. These facts would account for the small fluctuations in apparent dry weight that were observed. The uncertainty in the dry weight is, however, of little importance in the present investigation, as it cannot in any way affect the type of drying curve obtained; and its effect on the value of the constants must be small if not entirely negligible.

TABLE II

LOSS OF WATER FROM (PATENT) FLOUR SAMPLE 1A AT 25° C. OVER CONCENTRATED SULPHURIC ACID

Time	Weight Container + flour	Loss of weight	Percentage water remaining in flour (dry wt. basis)
min.	gm.	gm.	%
0	24.915	.....	13.065
15½	.878	0.037	11.93
40	.831	.084	10.48
51½	.810	.105	9.835
76½	.776	.139	8.79
86½	.765	.150	8.45
136½	.716	.199	6.95
153½	.705	.210	6.61
166½	.698	.217	6.40
187½	.688	.227	6.09
200½	.6836	.2314	5.96
215½	.6786	.2364	5.80
233½	.6710	.2440	5.57
251½	.6634	.2516	5.32
264½	.6580	.2570	5.17
281	.6514	.2636	4.97
332	.6330	.2820	4.40
347½	.6280	.2870	4.25
360½	.6240	.2910	4.125
387½	.6170	.2980	3.91
405	.6120	.3030	3.76
418½	.6086	.3064	3.65
429	.6060	.3090	3.56
448½	.6016	.3134	3.44
472½	.5970	.3180	3.30
491½	.5936	.3214	3.19
506½	.5914	.3236	3.12
574½	.5810	.3340	2.805
15 hr. (April 8)	.5250	0.3900	1.087
April 9	.5045		
15	.5030		
16	.4980		
17	.4955		
20	.4916		
21	.4920		
22	.4900		
24	.4900		
25	.4910		
28	.4930		
29	.4928		
30	.4940		
May 1	.4930		
2	.4922		
5	.4988		
6	.4998		
7	.4930		
8	.4920		
12	.4896*		
15	24.4900		

*Lowest wt. recorded in 39 days drying.
Greatest loss of weight recorded in 39 days drying = 0.4254 gm.
Weight of dried sample = 3.2572 gm.

the water contents are given in Figure 2 (Curve A). The first portion of the curve down to about 6.0% water is straight and on extrapolation would cut the horizontal axis at some point; the rate of drying over this portion of the curve is a linear function of the water content, i.e.— $\frac{dw}{dt} = k_1 (w - c_1)$*. The error involved in the graphical measurement of tangents is considerable, but by dividing each tangent obtained graphically in this portion of the curve by the corresponding value of w, we can obtain a mean value for k and from this value can calculate the tangents. Such calculated values agree fairly well with the observed ones and when checked by means of a straight-edge against the experimental curve from which the rates have been derived, are found to fit the curve quite as well as the observed values.

A stricter check, which has been applied in this particular case, is to integrate the above equation and to calculate times. Thus if $-\frac{dw}{dt} = k_1 (w - c_1)$ really is the equation representing the portion of the rate curve down to 6.0% water, the integrated form of this, i.e., $\log k'_1 (w - c_1) = k'_1 t + c$ is the equation of that portion of the experimental curve from which the rate curve has been derived. We therefore have

$$\log \frac{w_1 - c_1}{w_2 - c_1} = k'_1 t$$

in which w_1 and w_2 are any two values of w between 14% and 6.0% and t is the time taken for the moisture content of the sample to be reduced from w_1 to w_2. By taking two extreme values of w_1 and w_2, t for these values being read off from the large-scale experimental curve (Fig. 1), it is easy to calculate the value of k'_1; and by using this value all intermediate values of w can be calculated. This has been done and both calculated and observed values of w are given in Table III; the calculated points are shown graphically in relation to the experimental curve in Figure 1 (Curve A). It will be noticed how accurately the calculated points fit the smooth curve and how they fall away from it in the portion below 6.0%, where the equation ceases to apply. This affords a rigid proof that the portion of the rate curve considered is completely expressed by the equation given. The rate curve A, in Figure 2, is the calculated one that has been checked in this way; the calculated values of $-\frac{dw}{dt}$ are given in Table IV.

* A minus sign is employed as it is a negative gain, i.e., an actual loss of water, the rate of which is being measured.

Fig. 1.

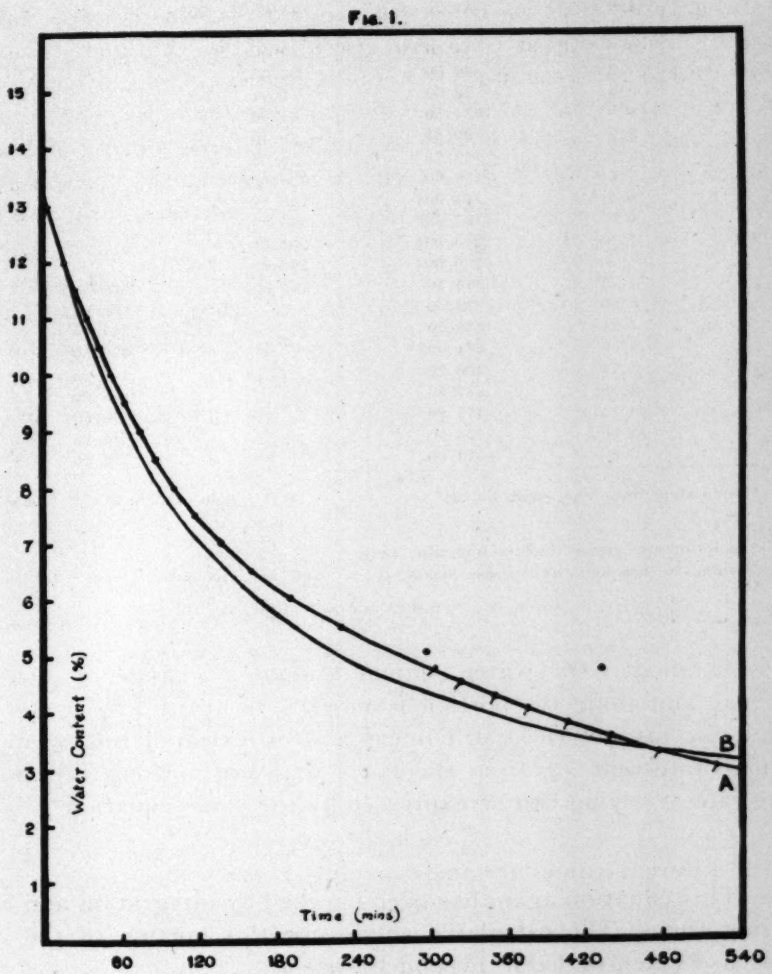

15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
Water Content (%)
Time (mins)
60
120
180
240
300
360
420
480
540
B
A

TABLE III

TIMES OF DRYING, OBSERVED AND CALCULATED (FLOUR SAMPLE 1A)

Water content	Time (observed)	Time (calculated)*	
%	min.	min.	
12.0	14.00	15.30	
11.5	22.50	23.00	
11.0	31.25	31.25	
10.5	40.00	40.13	
10.0	49.50	49.80	
9.5	60.50	60.35	
9.0	72.00	71.94	
8.5	84.50	84.90	
8.0	99.00	99.40	
7.5	116.25	116.28	
7.0	136.00	136.00	
6.5	161.00†	159.77	
6.0	197.50†	189.25	
5.5	240.00†	230.95	
5.0	279.00†	295.65	
4.75	300.50	431.50	(302.00)‡
4.50	322.50	.....	(322.50)‡
4.25	349.00	.....	347.75
4.00	374.00	.....	373.50
3.75	406.25	.....	405.75
3.50	437.50	.....	438.00
3.25	477.00	.....	476.50
3.00	524.00	.....	521.25
2.80	573.00	.....	573.00

*Calculated from the equation $\log \frac{w_1 - c_1}{w_2 - c_1} = k'_1 t$ in which $k'_1 = 0.003816$ and $c_1 = 4.31$.

†Experimental curve slightly irregular here.

‡Times in brackets and times below w = 4.50 are calculated from the equation $\log \frac{w_1 - c_2}{w_2 - c_2} = k'_2 t$, in which $k'_2 = 0.1092$ and $c_2 = 1.90$.

At about 6.0% water content a sudden change of direction occurs, and along the portion from 6.0% to about 2.5% water content, the rate curve is still linear and on extrapolation would cut off an intercept, c_2, from the horizontal axis. Along this portion the rate of drying can be expressed by the same equation

$$-\frac{dw}{dt} = k_2 (w - c_2)$$

with different values for the constants k and c.

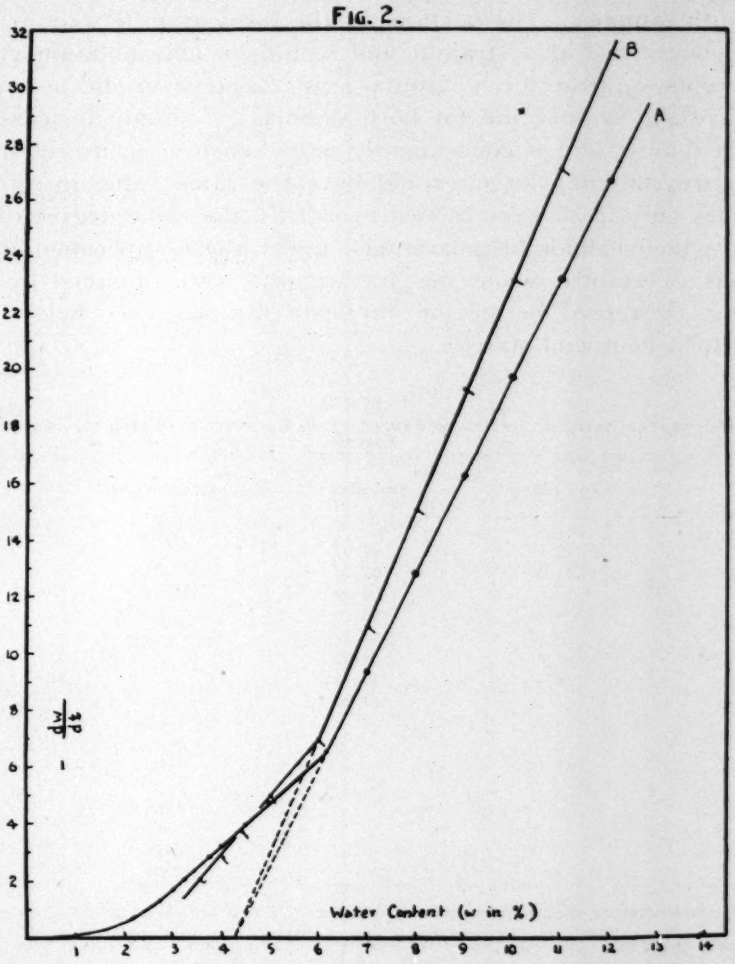
This equation again has been checked by integration and calculating times. The calculated values for this portion of the curve are also given in Table IV and Figure 2.

Below about 2.5% water content the rate curve bends round to the origin, drying becoming very slow. It is inferred that it is along this region that the really hygroscopic or sorbed water, or water that may perhaps be held chemically, is given off.

The determination was repeated on another sample of the same flour. Similar results were obtained and the experimental

data are shown in Table V and Figure 1, Curve B. In Table VI are given both observed and calculated times, the agreement between which shows that the same general equation holds with this sample as with the former. Calculated values for rates of drying ($-\frac{dw}{dt}$) at different moisture contents are given in Table VII and Figure 2, Curve B. Figure 2 shows that while the drying curves of the two duplicate samples are similar in type, being fitted by

FIG. 2.



the same general equation, the curves are not identical, i.e., the constant k in the equation $-\frac{dw}{dt} = k_1 (w - c_1)$, which measures the slope of the curve, has different values in the two cases. This is what would be expected. Containers of identical size and shape

were employed so that the area of drying surface is the same for both samples. The dry weights, however, and presumably also the closeness of packing of the flour particles and the thickness of the layers were different, so that identical rates of drying at identical water contents would not necessarily be observed. An interesting point is that the first straight portions of the rate curves, i.e., down to about 6.0% water, on extrapolation would pass through the same point on the horizontal axis; i.e., the constant c_1 has the same value for both samples. The portions of the rate curve between 6.0 and 2.5% water are also straight and would on extrapolation cut off intercepts, c_2, from the horizontal axis; c_2, however, did not appear to have the same value for both samples. C_2 is much nearer the origin than c_1 and is consequently more sensitive in its relation to moisture content; i.e., c_2 would have the same value in replicate samples only if all were carried to *exactly* the same degree of dryness, which cannot be guaranteed under these experimental conditions. Certainly, when the dry weights were obtained by oven drying, c_2 agreed better for duplicate samples (see below with Manitoba flour and starch).

TABLE IV

CALCULATED RATES OF DRYING OF SAMPLE 1A IN RELATION TO WATER CONTENT OF SAMPLE

Water content	$-\frac{dw}{dt}$ (calculated)*	$-\frac{dw}{dt}$ (calculated)†
%		
12.0	26.62	
11.0	23.14	
10.0	19.66	
9.0	16.19	
8.0	12.70	
7.0	9.22	
6.0	(5.74)	
5.0	(2.26)	(4.60)
4.5		3.92
4.25		3.56
4.00		3.16
3.75		2.78
3.50		2.42
3.25		1.94
3.00		1.64
2.75		1.26

*Calculated from the equation $-\frac{dw}{dt} = k_1 (w - c_1)$ in which $k_1 = 3.48$ and $c_1 = 4.3$. This held down to w = 7.0%, the curve being irregular between 6.75 and 4.75% w.

†Calculated from the equation $-\frac{dw}{dt} = k_2 (w - c_2)$ in which $k_2 = 1.51$ and $c_2 = 1.90$.

TABLE V

LOSS OF WATER FROM (PATENT) FLOUR SAMPLE 1B AT 25°C. OVER CONCENTRATED SULPHURIC ACID

Time	Loss of weight	Water remaining	Time	Loss of weight	Water remaining
min.	gm.	%	min.	gm.	%
0	.....	14.66	137½	0.252	6.92
8	0.025	13.99	152½	.264	6.55
15	.045	13.28	169	.276	6.18
22½	.067	12.60	183½	.285	5.90
30	.087	11.99	195	.291	5.72
36½	.102	11.53	213½	.300	5.44
49	.130	10.66	233½	.310	5.13
68½	.167	9.53	304	.335	4.365
90	.200	8.52	323	.340	4.21
101	.215	8.05	349	.346	4.03
113½	.230	7.59	386½	.354	3.78
124	0.240	7.85	436	.363	3.50
			506	.375	3.135
			22 hrs.	0.418	1.506

Greatest loss of weight in 37 days drying = 0.477 gm.
Weight of dried sample = 3.253 gm.

TABLE VI

TIMES OF DRYING, OBSERVED AND CALCULATED (FLOUR SAMPLE 1B)

Water content	Time (observed)	Time (calculated)*
%	min.	min.
12.0	30.0	30.0
11.5	36.5	36.40
11.0	43.75	43.75
10.5	51.25	51.40
10.0	60.00	59.70
9.5	69.00	68.75
9.0	78.75	78.80
8.5	89.25	89.90
8.0	101.25	102.30
7.5	116.25	116.75
7.0	134.75	133.50
6.5	155.00	153.80
6.0	179.5	179.25
5.75	193.00	(194.80)†
5.50	207.5	213.65
5.00	243.00	266.85
4.75	263.50	310.50
4.50	288.00	390.70
4.40	300.00	459.20
4.00	353.00	
3.75	392.50	
3.50	437.5	
3.25	485.00	

*Calculated from the equation $\log\frac{w_1 - c_1}{w_2 - c_1} = k'_1 t$, in which $k'_1 = 0.0044$ and $c_1 = 4.3$. This equation holds from $w = 12.25\%$ to $w = 6.00\%$ inclusive.

†Break in rate curve occurs here, hence equation ceases to apply without change in values of constants.

TABLE VII

CALCULATED RATES OF DRYING OF SAMPLE 1B IN RELATION TO WATER CONTENT OF SAMPLE

Water content	$-\frac{dw}{dt}$ (calculated)*	$-\frac{dw}{dt}$ calculated by arithmetical method and × 396	Difference in $-\frac{dw}{dt}$ as determined by the two methods
12.0	31.20		
11.0	26.88	26.87	−0.01
10.0	22.86	22.32	−0.54
9.0	18.85	19.58	+0.73
8.0	14.84	14.73	−0.11
7.0	10.83	10.26	−0.57
6.0	6.82	7.56	+0.74
5.5	....	6.26	
5.0	(2.80)	4.94	
4.5	....	3.64	
4.0	....	2.73	

*Calculated from the equation $-\frac{dw}{dt} = k_1 (w - c_1)$ in which $k_1 = 4.0125$ and $c_1 = 4.3$. This equation holds from w = 12.0% to w = 6.0% inclusive.

The idea suggests itself that the intercepts c_1 and c_2 cut off from the horizontal axis by extrapolating the straight portions of the rate curves may be different for different flours. The values of c_1 and c_2 may be characteristic of a flour, or may be correlated in some way with quantity and/or quality of gluten. It is known that gluten holds water far more tenaciously than does starch, while quality of gluten may come in also as a factor.

To test this point, determinations of rates of drying were carried out on two samples (2A and 2B) of a No. 1 Northern Manitoba straight run flour which had a higher gluten content than the City Imperial. The experimental results are given in Table VIII. The data were analysed as before, altho a somewhat different method was employed (see p. 202), and similar equations of rate were found to apply. The calculated values for rates of drying $(-\frac{dw}{dt})$ are given in Table IX and shown graphically in Figure 3. The points do not lie quite so close to the straight rate curve as was the case with Samples 1, A and B, but are quite close enough to confirm the linear relationship between rate of drying and water content.[2] With these samples the duplicate curves are substantially identical. although this identity is probably purely accidental.

2 The slight irregularities are probably due to the fact that the arithmetical method of calculating the tangents is not so accurate as the method used for the earlier samples; slight irregularities in the drawing of the large scale experimental curve are magnified in calculating the rates. The experimental curve for sample 1A became also distinctly irregular at w = 6.5%, but became normal again at w = 5.0%. Such irregularities are possibly due to internal movements and re-distributions of flour particles under the influence of stresses caused by the tensions of the fine capillary water columns. cf. Fisher (1924b, 1924c).

FIG. 3.

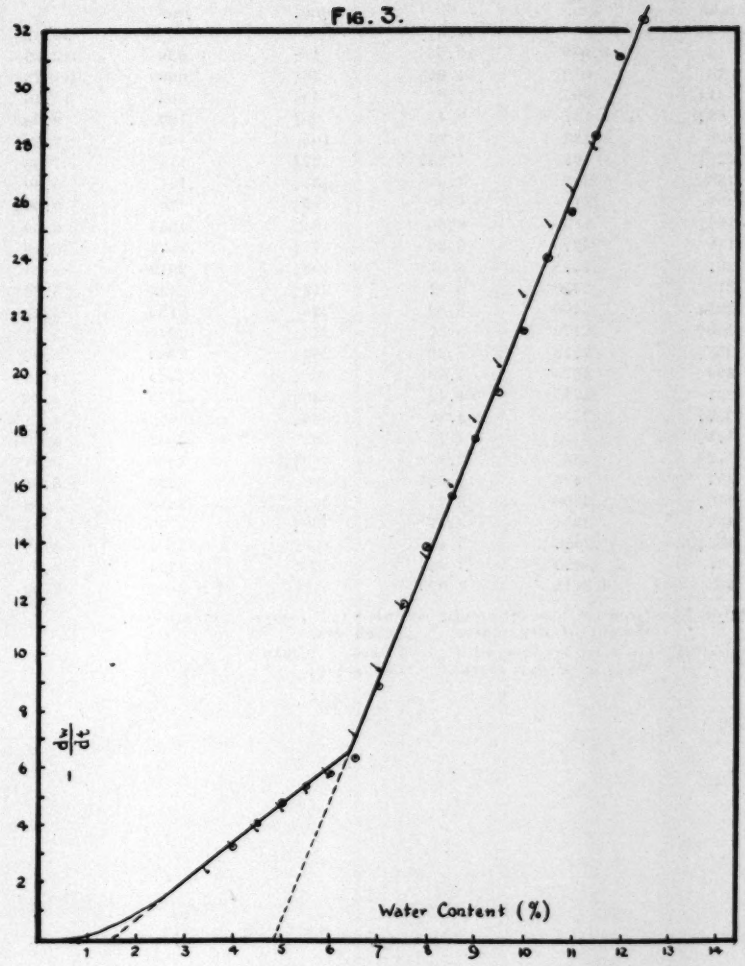

32
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
$-\frac{dx}{dt}$
1
2
3
4
5
6
7
8
9
10
11
12
13
14
Water Content (%)

TABLE VIII

LOSS OF WATER FROM NO. 1 MANITOBA FLOUR (SAMPLES 2A AND 2B) AT 25°C. OVER CONCENTRATED SULPHURIC ACID

Sample 2A			Sample 2B		
Time	Loss of weight	Percentage water remaining	Time	Loss of weight	Percentage water remaining
min.	gm.	%	min.	gm.	%
0	.....	14.53	0		13.49
15	0.040	13.05	11¼	0.030	12.39
28	.070	11.955	26¼	.060	11.28
41¼	.095	11.0	39¼	.082	10.46
68¼	.137	9.45	65¼	.120	9.06
108	.180	7.85	105	.161	7.54
125¼	.195	7.297	122¼	.174	7.06
138	.203	7.00	134¼	.181	6.80
151	.211	6.70	149¼	.189	6.505
165¼	.2165	6.50	164¼	.1945	6.30
179	.2210	6.33	177¼	.1975	6.19
201	.2295	6.015	198¼	.2070	5.84
213	.2350	5.81	212¼	.2125	5.62
227¼	.2400	5.63	225	.2185	5.415
246¼	.2470	5.37	243¼	.2240	5.21
258¼	.2515	5.20	254¼	.2280	5.06
309	.2675	4.605	306¼	.2435	4.49
323	.2715	4.45	320¼	.2470	4.36
338¼	.2755	4.31	336¼	.2510	4.215
354¼	.2792	4.17	351	.2542	4.095
372¼	.2835	3.975	369¼	.2585	3.94
393	.2874	3.865	390	.2620	3.81
410¼	.2908	3.74	407¼	.2655	3.68
435	.2954	3.57	430¾	.2700	3.51
455¼	.2986	3.45	451¼	.2730	3.40
534	.3090	3.065	530¼	.2834	3.015
553	0.3114	2.975	548¾	0.2860	2.92

Sample 2A—Greatest loss of weight recorded in 73 days = 0.3916 gm.
Weight of dry matter = 2.6589 gm.

Sample 2B—Greatest loss recorded in 73 days = 0.3650 gm.
Weight of dry matter = 2.6455 gm.

TABLE IX

CALCULATED RATES OF DRYING OF NO. 1 MANITOBA FLOUR (SAMPLES 2A AND 2B) IN RELATION TO WATER CONTENT

Water content	Rate = $-\frac{dw}{dt}$	
	Sample 2A	Sample 2B
%		
14.0	40.66	.....
13.5	38.05	.....
13.0	35.27	36.12
12.5	32.35	32.99
12.0	31.09	31.12
11.5	28.35	27.85
11.0	25.66	26.41
10.5	24.04	25.19
10.0	21.46	22.70
9.5	19.32	20.31
9.0	17.66	18.25
8.5	15.72	16.04
8.0	13.94	13.46
7.5	11.78	11.52
7.0	8.59	9.35
6.5	6.35	7.09
6.0	5.70	5.66
5.5	5.36	5.07
5.0	4.73	4.55
4.5	4.01	3.79
4.0	3.19	3.15
3.5	2.33	2.365

Similar determinations were carried out on samples of wheat starch (nitrogen content = 0.05%) and of crude gluten (nitrogen content = 7.75%; N × 5.7 = 44.18% crude protein), details for which are given in Tables X and XI. Rate curves are shown in Figures 4 and 5. It will be seen from an examination of these four sets of data that while the rate-of-drying curves are all of the same type, the values of the constants c_1 and c_2 increase with (but not necessarily as) the gluten or crude protein content of the flour. It might be expected in this case that the position of the point of intersection of the two linear portions of the rate curve would also depend on the gluten content, since the latter would affect very considerably the length of the curved portion of the rate curve immediately to the right of the origin. The method of work is probably not sufficiently accurate to show this. The equation of rate contains two constants, k and c, and a very slight alteration in one of them can be compensated for by a slight alteration in the other without any serious error in the calculated values of $-\frac{dw}{dt}$. Such a slight alteration in k, however, would affect appreciably the value of w at which the point of intersection occurs.

TABLE X

CALCULATED RATES OF DRYING OF WHEAT STARCH (SAMPLES 3A AND 3B) IN RELATION TO WATER CONTENT

Water content	Rate = $-\frac{dw}{dt}$	
	Sample 3A	Sample 3B
%		
15.0	41.70	.....
14.5	38.75	36.00
14.0	35.24	33.80
13.5	33.73	31.68
13.0	32.36	30.56
12.5	30.45	27.34
12.0	28.90	26.40
11.5	26.40	25.60
11.0	23.40	22.35
10.5	19.37	19.37
10.0	18.00	18.00
9.5	17.25	17.25
9.0	15.71	15.71
8.5	14.06	14.06
8.0	12.60	12.60
7.5	11.42	11.42
7.0	9.65	9.65
6.5	8.06	8.105
6.0	6.62	6.90
5.5	5.06	5.64
5.0	4.165	4.52
4.5	3.545	3.85
4.0	3.00	3.29
3.5	2.28	2.635
3.0	.....	2.13

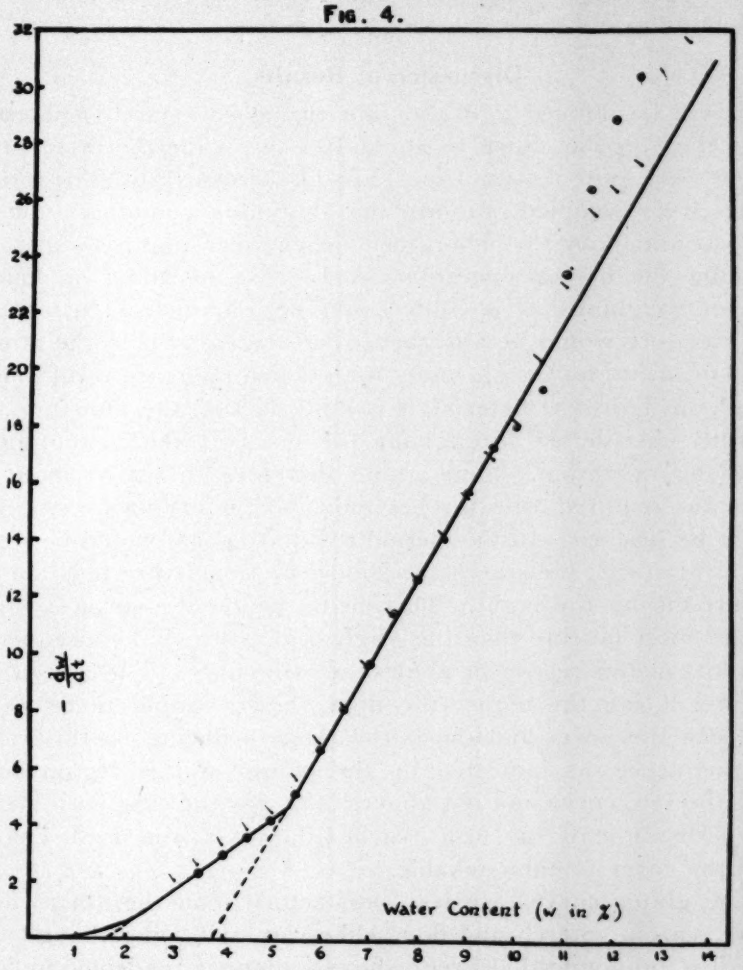
TABLE XI

CALCULATED RATES OF DRYING OF CRUDE GLUTEN (SAMPLES 4A AND 4B) IN RELATION TO WATER CONTENT

Water content	Rate = $-\frac{dw}{dt}$	
	Sample 4A	Sample 4B
%		
13.5	.....	23.30
13.0	24.43	21.76
12.5	22.45	20.86
12.0	21.71	20.06
11.5	21.03	19.56
11.0	19.83	18.87
10.5	18.85	17.63
10.0	18.44	15.87
9.5	17.03	14.55
9.0	15.41	13.23
8.5	14.16	11.67
8.0	12.52	10.19
7.5	10.66	8.73
7.0	9.095	7.36
6.5	7.64	6.205
6.0	6.11	5.195
5.5	4.965	4.28
5.0	4.275	3.53
4.5	3.645	2.87
4.0	2.03	2.19
3.5	1.335	1.515

The moisture content of the No. 1 N. Manitoba flour used in this work was determined immediately after samples 2A and 2B had been weighed out. The test was carried out in a Hearson electric oven at 120°C. in the manner usual in the writer's laboratories,[3] and the results were: 13.75, 13.75, 13.78, 13.71, 13.74, 13.77%; mean value 13.75% on the usual air-dry basis, which equals 15.94% on a dry weight basis. The probable error (PE_s) of a single determination under these conditions was found to be $\pm$ 0.06%[4]. A deviation from the mean value of $\pm$ 0.18% (i.e. $PE_s \times 3$) cannot, therefore, be regarded as significant.[3] The total moisture content

FIG. 4.



[3] Research Ass'n British Flour Millers, Confidential Report No. 1.

[4] Based on a large number of determinations; see Research Ass'n British Flour Millers Confidential Report No. 1, (1925).

found in the above experiments over sulphuric acid at 25°C. was 16.06% for sample 2A, and 15.70% for sample 2B. The first is well within three times the PE_s from the electric oven figure, while the latter is just beyond it. The electric oven figure therefore appears to be sufficiently accurate for this kind of work and by adopting it a great saving in time results. It is not necessary to take daily weighings for a month or so in order to obtain the dry weight, as this can be obtained with sufficient accuracy from the electric oven figures. The latter figure was employed in calculating the dry weight of the starch and crude gluten used in this investigation.

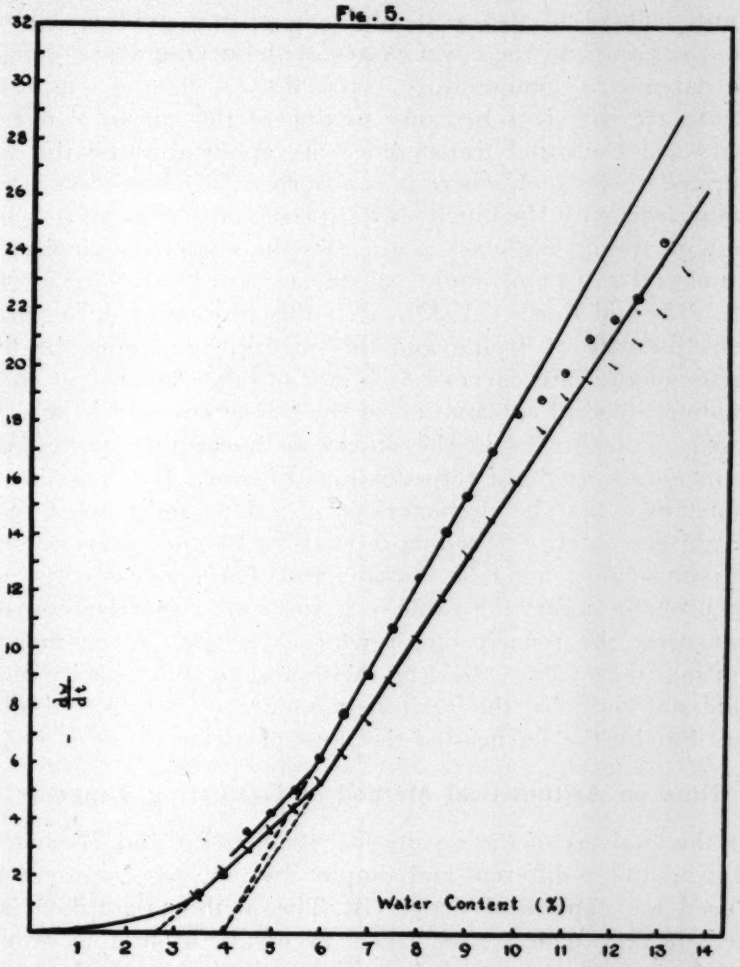
Discussion of Results

It will be noticed that the rate curves for starch and crude gluten are irregular down to about 10% w, while the rate curves for flour were quite regular from 13 or 14% downward. The former samples were weighed out into the aluminum containers and allowed to stand on the laboratory bench over night so that, by beginning the drying operations early next morning, as long a series of weighings as possible could be obtained. During the night moisture would be absorbed from or given up to the atmosphere but, owing to the slowness with which moisture would move through such air-dry material, it is unlikely that the moisture was uniformly distributed throughout the mass at the beginning of the drying operation. Some time, therefore (actually about an hour), was required after the beginning of the drying for a steady state to be reached. In this period the drying rate would be somewhat erratic, e.g., the starch (as shown by a moisture test) gained moisture during the night. This, being nearer the surface, would come off more quickly than the original moisture and consequently the points in this region lie above the rate curve. The gluten lost moisture during the night; the upper layers would therefore be drier than the lower and the initial rates of drying would be low in consequence, as indicated by the points in this region being below the rate curve and not above it, as was the case with starch. Once a steady state has been reached, however, the linear character of the curve is unmistakable.

The gluten curves are less satisfactory than the others for a further reason. Starch and flour (like clay and soil although to a much less pronounced degree) show a distinct tendency to form a coherent mass during drying—a mass sufficiently coherent to resist the disturbing effect of thermostat movements. This par-

ticle-coherence is probably caused by the very high tensions that must exist in the minute water wedges[5] between the particles, in consequence of which the particles are pulled closely together and tend to stick even after all the water has been lost by evaporation. The gluten, however, was very flocculent with no tendency to stick, and it was found impossible to avoid considerable disturbances within the gluten powder during drying due to rotation of thermostat; in spite of these difficulties, the linear character of the rate curves is unmistakable, altho there is a rather bad kink in Curve A, Figure 5, between 4 and 4.5% w.

FIG. 5.



5 These tensions may equal 1000 or 1200 atmospheres at very low water contents; see Fisher (1924b, 1924c), and Whipple (1922).

It should also be pointed out that the temperature of the drying mass is not that of the thermostat (25°C.), but lower. It is obvious that a mass of moist or air-dried material kept in a dry atmosphere is under conditions comparable to those of a wet-bulb thermometer. At 25°C. and relative humidity 0, the temperature of a wet-bulb thermometer would fall rapidly to about 8.5° C., as the wet-bulb depression for these conditions is 16.5°C. It will be shown elsewhere[6] that the actual temperature of the drying material at the wet end of the curve is 9.5 to 10°C. This temperature rises steadily as drying proceeds to 21.5°C., at which it remains constant. There is also a steady temperature gradient between the acid (25°C.) and the cover plates of the drying vessel (slightly above laboratory temperature, say 20°C.). These temperature gradients are sufficient to cause mixing of the air by convection currents and the rapid transference of vapour through the air to the surface of the acid, where it is absorbed. This process is rapid in comparison with the much slower process of diffusion of vapour throughout the drying mass and across the stationary air film that surrounds all solid and liquid substances, as shown by Langmuir (1912, 1913) and Lewis (1922). It is this process of diffusion that controls the rate of drying and this probably explains the linear character of the rate curves. It is not possible at present to give a complete physical explanation of the whole course of the drying process, of the breaks in the otherwise linear rate curves, or of the constants k and c of the equations of rate. It is possible that the constant c_2 may be characteristic of a flour and may be related to the gluten content. Much more work will be necessary to decide these points, as it must be remembered that fineness of grinding would probably affect the value of c; after all, c merely represents, or measures, the tenacity with which the last of the moisture, amounting to 2 or 3%, is held by the flour, and this tenacity may be affected not only by the relative amounts of starch and gluten present but by the fineness of the flour particles.

Note on Arithmetical Method of Evaluating Tangents

For the analysis of the results for Samples 2A and 2B and subsequent results, a different and simpler method was employed than that used for Samples 1A and 1B. The method described is accurate but very tedious; its use is necessary at first in order to establish the general type of equation applicable to the curves. For subsequent work the arithmetical method described below is

[6] J. Agr. Sci. 1927; J. Text. Inst. 1927.

sufficiently accurate provided the large-scale curves are very carefully and accurately drawn. This arithmetical method of calculating tangents is new and has not hitherto been published. It was devised by Dr. R. A. Fisher, chief statistician at the Rothamsted Experimental Station. Given a set of values of w and t, the values are plotted on a large scale and t equivalent to each % w is read off by interpolation. Values for the *reciprocals* of the tangents can be obtained from these data by means of the equation

$$\frac{dt}{dw} = t_1 - t_0 - \frac{1}{24}(\Delta^2 t_1 - \Delta^2 t_0)$$

This equation is applied as follows: in Table XII values of w differing by 0.5% and corresponding values of t are given in the first two columns.[7] In the third column are given the first differences, i.e., the difference between each value of t and the preceding value. Each first difference, therefore, represents the time taken for the loss of 0.5% water at a given water content. In the fourth column are given the sums of each successive pairs of first differences, i.e., $t_1 - t_0$, which represents the time taken for the loss of 1% of water. In the fifth column are given the second differences, i.e. the differences between each first difference and the preceding value, or $\Delta^2 t_1 - \Delta^2 t_0$. In the sixth column are given the values of $\frac{1}{24}(\Delta^2 t_1 - \Delta^2 t_0)$; these values subtracted from the values of $t_1 - t_0$ (of the fourth column) give values for $\frac{dt}{dw}$ which are shown in the seventh column. Finally, reciprocals of $\frac{dt}{dw}$ are the values of the tangents, $\frac{dw}{dt}$, given in the eighth column, while the latter values multiplied by 396 for comparison with corresponding figures in Table VII and for convenience of plotting, are given in the ninth column. It is important that the difference in w corresponding to $t_1 - t_0$ should be the same throughout the table. In the present work 1% has been taken for this "span." It is sometimes necessary at the "dry" end of the curve to make the "span" less than unity, say 0.5 or 0.25; this would make $\frac{dt}{dw}$ of column 7 half or a quarter of its proper value. Similarly, if at the "wet" end of the curve a "span" of 2 or 4 were employed, the corresponding value of $\frac{dt}{dw}$ would be doubled or quadrupled and the necessary and obvious arithmetical correction would have to be made. Further, if a change of span is employed, overlap should be arranged over a region in which the change occurs so as to avoid artificial kinks in the curve. As a rule, however, it is not difficult to avoid such changes of span.

[7] These are the data for flour Samples 1B given in Table VI.

The kind of agreement obtained between this comparatively simple arithmetical method and the stricter though more laborious method based on integration is shown in the third and fourth columns of Table VII.

TABLE XII

ILLUSTRATING ARITHMETICAL METHOD OF CALCULATING TANGENTS TO AN EXPERIMENTAL CURVE

Data for Flour Sample No. 1B from Table VI

Water	Time	First difference	$t_1 - t_0$*	Second difference $\Delta^2 t_1 - \Delta^2 t_0$§	Second difference / 24	$\frac{dt}{dw}$†	$\frac{dw}{dt}$‡	$\frac{dw}{dt} \times 396$
%	min.							
12.5	23.5							
		6.5						
12.0	30.0		13.0	0.0	0.00	13.00	0.07692	30.45
		6.5						
11.5	36.5		13.75	0.75	.03	13.72	.07288	28.86
		7.25						
11.0	43.75		14.75	0.25	.01	14.74	.06784	26.87
		7.5						
10.5	51.25		16.25	1.25	.05	16.20	.06173	24.44
		8.75						
10.0	60.0		17.75	0.25	.01	17.74	.0564	22.32
		9.0						
9.5	69.0		18.75	0.75	.03	18.72	.0534	21.15
		9.75						
9.0	78.75		20.25	0.75	.03	20.22	.0494	19.58
		10.5						
8.5	89.25		22.5	1.5	.06	22.44	.0445	17.65
		12.0						
8.0	101.25		27.0	3.0	.12	26.88	.0372	14.73
		15.0						
7.5	116.25		32.5	2.5	.10	32.40	.0308	12.22
		17.5						
7.0	133.75		38.75	3.75	.15	38.60	.0259	10.26
		21.25						
6.5	155.0		45.75	3.25	.13	45.62	.0219	8.68
		24.5						
6.0	179.5		52.5	3.5	.14	52.36	.0191	7.56
		28.0						
5.5	207.5		63.5	7.5	.28	63.22	.0158	6.27
		35.5						
5.0	243.0		80.5	9.5	.40	80.10	.0125	4.95
		45.0						
4.5	288.0		110.0	20.0	.83	109.17	.0091	3.63
		65.0						
4.0	353.0		149.5	19.5	0.82	148.68	0.0067	2.66
		84.5						
3.5	437.5							

*$t_1 - t_0$ = sum of 1st differences.

†$\frac{dt}{dw} = t_1 - t_0 - \frac{1}{24}(\Delta^2 t_1 - \Delta^2 t_0)$

‡$\frac{dw}{dt}$ = reciprocal of $\frac{dt}{dw}$.

§$\Delta^2 t_1 - \Delta^2 t_0$ = difference of 1st differences.

Summary

A study has been made of the rates of drying of flour, starch, and crude powdered gluten. The experimental method consisted in exposing the flour over concentrated sulphuric acid in a closed vessel at a constant temperature; and the apparatus was so arranged that the flour could be weighed at intervals without removing it from the drying vessel or in any way disturbing the drying process or the drying conditions.

When rates of drying ($-\frac{dw}{dt}$) are plotted against moisture content (w) the rate curves are all of the same type and consist of three parts separated by more or less sudden changes of direction. Down to 6% w the curve is linear and on extrapolation would cut off an intercept, c_1, from the horizontal axis. At about w = 6% a sudden change of direction occurs and between 6% and 2½% the rate curve is again linear and on extrapolation would cut off an intercept, c_2, from the horizontal axis, c_2 being less than c_1. Along these two linear portions of the curve the rate of drying can be accurately expressed by the equations:

$$-\frac{dw}{dt} = k_1 (w - c_1) \qquad (1)$$

$$-\frac{dw}{dt} = k_2 (w - c_2) \qquad (2)$$

There is some evidence to suggest that c_1 (and possibly c_2) may be characteristic of each flour and may be connected with the amount of gluten present in the flour. Below w = 2.5%, the rate curve bends round to the origin, drying becoming extremely slow. It is inferred that along this portion of the curve the really hygroscopic moisture is given off.

These rate curves are essentially similar in type to those obtained for other materials, e.g., textile fabrics, soils, and clays.

An explanation, based on considerations of diffusion of vapour through the material, is suggested.

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NOTES ON THE USE OF OSTWALD VISCOMETERS FOR FLOUR SUSPENSIONS

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The capillary bore tube viscometer developed by Wilhelm Ostwald, and employed by Wolfgang Ostwald and Lüers in their original researches on the physico-chemical properties of flour suspensions, has been somewhat neglected in recent years by cereal chemists in favour of rotating cylinder instruments such as the Couette, the MacMichael, and the Stormer. This is partly due, no doubt, to the criticisms made by Sharp and Gortner (1923). These authors found the Ostwald viscometer difficult to use on account of a gradual "fall back" in the time of flow of a given flour suspension, which made readings of doubtful value. The present authors have found that this "viscosity fall back" when extrapolated to zero time, is a constant for any given flour, and partially at least, correlated with the "strength" of that flour. It has also been found that Ostwald viscometers of widely different bore, when properly calibrated with liquids of known viscosity, give strictly comparable results on flour suspensions, provided that these results are expressed as actual viscosities and not merely as times of flow relative to water (a practice recommended in certain textbooks, which has been found to give very misleading results); provided always that the viscometers used are so designed as to give laminar or stream line flow at the viscosities at which they are to be used.

The Ostwald viscometer possesses certain advantages over the rotating cylinder types in that it is cheap, easily constructed by any glassblower from stock laboratory tubing, and of dimensions varying between wide limits without affecting accuracy of the results. A considerable number may be used in a small space simultaneously, while temperature control (which in the rotating cylinder type requires a constant temperature room, or elaborate jacketing) is effected very simply by the use of a thermostat tank of simple pattern.

Theory

The determination of the viscosity of a liquid by means of the Ostwald viscometer depends on the fact that liquids flowing through a pipe at any velocity below a certain critical value obey Poiseuille's Law, which, in its simplest form may be written:

$$(1) \quad V = \frac{\pi (p_1 - p_2) a^4}{8 \eta h}$$

where

V is velocity of flow,

p_1, p_2 are pressures at upper and lower ends of the capillary,

a is the radius,

h is the length of the capillary, and

η the viscosity of the liquid.

From this may be deduced the formula

$$(2) \quad \eta = Kt\rho$$

where

K is a constant for each viscometer,

t is the time of fall of liquid, and

ρ the density thereof.

In practice it is found that the relationship is rather more complex than this, and various errors may be introduced by acceleration periods, kinetic head, finite values of the function h/a, and other factors not covered by the basic equation. Various corrections, chiefly empirical, have been suggested from time to time to neutralise these errors, but have not been entirely satisfactory and may be disregarded.

On plotting $\eta/t\rho$ it will be found that this is not a straight line curve, and that in practice it does not pass through the origin. For these reasons the common practice of taking the ratio t(liquid): t(water) as a measure of relative viscosity gives an entirely incorrect value; and to obtain approximately correct results it is necessary to calibrate each viscometer individually throughout the

range of its application with liquids of known viscosity, such as those given by Green (1908). This procedure depends on the assumption that V, in the range over which the viscometer is calibrated, is below the critical value mentioned above, and that the flow in the capillary is laminar and not turbulent, since in the latter case there is at present no known relation between η and t, although it is understood that work is in progress in this field. It should be pointed out, however, that although the flow in a given tube may be turbulent with water, it will not necessarily be so with liquids of higher viscosity, which makes it possible to use wide-bore viscometers with their attendant advantages on flour suspensions of high concentration. The viscometer having been calibrated with liquids of known viscosity and density, it is, in many cases, justifiable to neglect the density of flour suspension when a result of no great accuracy is required, since the density varies only slightly over a relatively wide range.

Two equations are given by Osborne Reynolds (1883) for the determination of the critical velocity V_c

$$(3) \quad V_c = \frac{114.2}{a\,f}$$

where f is the reciprocal of the viscosity relative to water at 0°C. and

$$(4) \quad V_c = \frac{1000\,\eta}{\delta\,a}$$

where η is the viscosity in absolute units. In practice it is convenient to take the centipoise ($\eta \div 100$) as the unit of measurement. The values given by (4) are rather lower than those found by direct experiment, and the inclusion of the density factor makes this expression more reliable for practical purposes. The largest bore viscometer used in the work quoted below was given a safe critical velocity for water by (3) and an unsafe velocity by (4). Actually it was found by direct experiment, using the method of Reynolds (loc. cit.) and also by the position of the water time point on the standardisation curve, that the velocity was on the safe side of the critical value.

The figures recorded in Table I, taken from Green's tables, may be of use for the calibration of viscometers.

TABLE I

GREEN'S TABLES FOR THE CALIBRATION OF VISCOMETERS

Viscosity in absolute units η	Normality of sugar solution n	$\log \left[\frac{\log \eta/\eta_0}{n}\right]$
0.008953	0	—
.010431	0.1723	$\bar{1}.5849$
.011541	0.2779	$\bar{1}.5983$
.012903	0.3875	$\bar{1}.6120$
.017602	0.6603	$\bar{1}.6479$
.019088	0.7242	$\bar{1}.6569$
.020914	0.7947	$\bar{1}.6661$
.026387	0.9629	$\bar{1}.6878$
.033453	1.1205	$\bar{1}.7088$
.04997—	1.3472	$\bar{1}.7386$
.07810—	1.5905	$\bar{1}.7719$
.13280—	1.8255	$\bar{1}.8072$
.2388— —	2.0517	$\bar{1}.8420$
0.4658— —	2.271—	$\bar{1}.8780$

Where η_0 is viscosity of water at 25° C.

The interpolations from these figures are obtained by plotting log. $\left[\frac{\log \eta/\eta_0}{n}\right]$ against n and in this way the curve will approximate to a straight line.

Viscometer design.—Various patterns of viscometer have been put forward as being suitable for flour suspensions. The tubes used by Ostwald and Lüers had the following dimensions:

Capillary tube 15-20 cm. long, temperature 20°C., water time about 2 minutes, bulb capacity 5-10 cc. That used by Sharp and Gortner had a bulb of about 23 cc., a capillary of about 9 cm. length, and 0.85 mm. bore, having a water time of 9.2 seconds at 25°C.

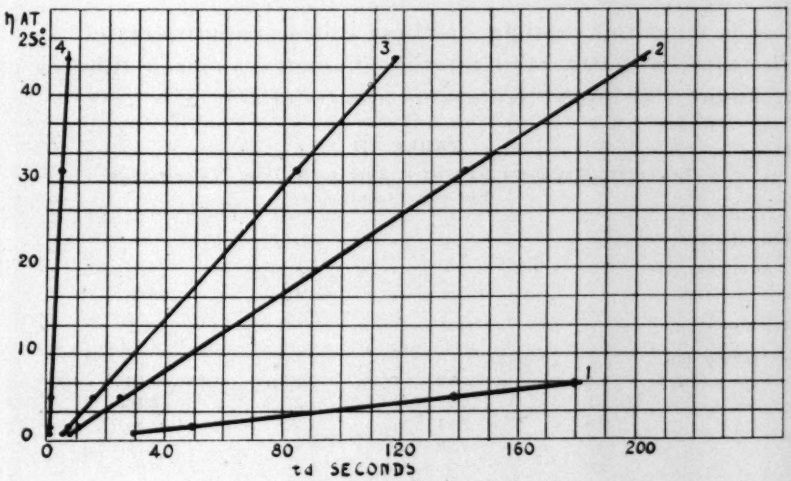

Fig. 1. Viscosity Calibration Curves

It has been found in this laboratory that the dimensions of the viscometer, if properly calibrated and used, have little or no effect on the results (so long as these are expressed in absolute units), with flour suspensions below 30%, at which figure slight irregularities begin to occur in some tubes, though it is not yet clear whether these are due to plasticity effects or to pre-gel formation, already discussed (1927), which are possibly aspects of the same phenomenon. It has been found that no advantage is gained by having a second smaller bulb below the upper, as in some viscometers—notably those recommended by the British Engineering Standards Association (1923)—in order to give, apparently, a suitable funnel shaped opening to the capillary. It has, however, been found that the tube above the lower bulb should be as wide as possible to avoid difficulties due to frothing (which may be serious when viscosities are measured at pH other than the normal flour range), and that the tube should not be greatly constricted at the upper zero mark. (Calibration curves for these tubes are given in Figure 1. Unless otherwise stated, all viscosity measurements described in this paper were made at 25°C.)

TABLE II

CALIBRATION OF OSTWALD VISCOMETER CAPILLARY TUBES

Tube No.	Capillary bore	Water time	Average water velocity
	cm.	sec.	sec.
1	0.138	29.0	46
2	.218	7.4	73
3	.238	5.2	87
4	0.496	1.0	103

The length of capillary in these tubes is approximately 20 cm., bulb capacity 20 cc., and internal diameter of back tube 15 mm. The amount of liquid required is approximately 25 cc.

TABLE III

VISCOSITY MEASUREMENTS WITH DIFFERENT BORE VISCOMETERS

Viscosity (centipoises)

Viscometer No.		1	2	3	4
Bore of viscometer (cm.)		0.138	0.218	0.238	0.496
Water time (sec.)		29.0	7.4	5.2	1.0
Percentage of flour in suspension	2.5	1.02	0.97	1.02	.....
	5	1.22	1.22	1.16	.....
	10	2.24	2.17	2.20	.....
	15	4.20	3.88	4.05	4.10
	20	.....	8.00	8.00	8.30
	25	.....	23.30	22.90	23.40

Section 2. Viscosity Fall Back

Sharp and Gortner (1923), as already stated, found some difficulty in measuring the viscosity of flour suspensions in the Ostwald viscometer on account of the tendency of the apparent viscosity to decrease with time, and it appears from their paper that similar effects were obtained with the MacMichael viscometer, since their technique involved the taking of measurements at one hour after making up the suspensions to obtain comparative results.

The variation of apparent viscosity with time might be expected to involve three factors:

(a) It may depend on a change in the viscosity due to repeated shearing (as in the case of gelatin and silicic acid sols, described by Garrett (1903) and others).

(b) It may be due to a colloidal change actually taking place within the suspension, e.g., in gelatin there is an increase of viscosity with time and in ceric hydroxide a decrease. (Hatschek, 1922).

(c) There may be chemical changes occurring, such as proteolysis, owing to the presence of enzymes and other catalysts.

Investigation of the fall back of flour suspension viscosities showed that, subject to reasonable stirring at intervals, the phenomenon is independent of the number of times of shearing in the viscometer, two identical suspensions falling back to the same extent over a period of some hours, when one was sheared twice as many times as the other. Further, the phenomenon is not due to the influence of proteolytic enzymes, since 'weak' flours showing high

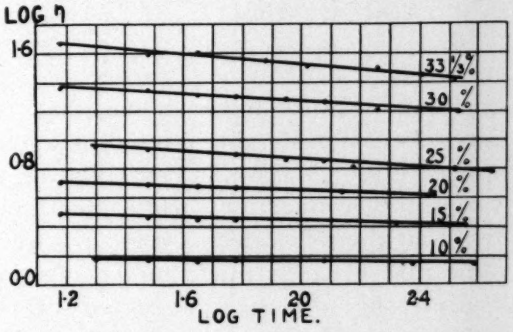

Fig. 2. Viscosity : Time for Various Concentrations

proteolytic activity as measured by the method described by Denham and Scott Blair (1927), show, normally, rather low fall backs, whereas, 'strong' flours with low proteolytic activity show high fall back; and maintaining the flour for some time at a temperature sufficient to abolish proteolysis does not appreciably affect the slope of the fall back curve.

In order to obtain a figure for the fall back of a given flour suspension, the logarithmic viscosity (absolute) is plotted against the logarithmic time, and a straight line results. If this straight line slope is plotted against the concentration of the flour, another straight line is obtained, and that the error of the points on this line is quite independent both of the number of times of shearing and of the dimensions of the viscometer used, adds additional support to the above conclusions. These results are shown in Tables IV and V, the curves being numbered 2 and 3.

In the final curve, No. 3, the appended figures indicate the number of the viscometer used and the number of times of shearing during the whole run.

If the explanation of viscosity fall back must be sought in a colloidal change taking place within the suspension, it is apparent that similar changes may be expected to occur in other colloidal systems.

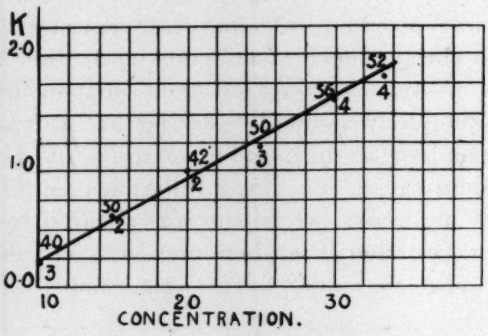

Fig. 3. Fall-Back Slope : Concentration

Hatschek and Jane (1926) have shown that the elasticity modulus of various sols is considerably altered by ageing, and have discussed the applicability of Maxwell's law (relating this function to the viscosity) in this connection, while Biltz and Vegesack (1910) have shown that there is a decrease in the viscosity of solutions of night blue and benzopurpurin in ageing, and that this phenomenon is considerably affected by repeated shearing. Ostwald (1913) and Zsigmondy (1922) have also discussed the connection which exists between these changes in viscosity and the colloidal structure of the substances investigated.

TABLE IV

VARIATION OF VISCOSITY FALL-BACK WITH CONCENTRATION

Flour D

Viscometer No.	Viscometer bore	Concentration	Time	log time	η	log η
		%	min.			
3	0.238	10	20	1.30	1.50	0.18
			30	1.48	1.50	0.18
			45	1.65	1.45	0.17
			60	1.78	1.45	0.17
			120	2.08	1.45	0.17
			240	2.38	1.40	0.15
			390	2.59	1.40	0.15
2	0.218	15	15	1.18	3.10	0.49
			30	1.48	2.90	0.46
			45	1.65	2.85	0.45
			60	1.78	2.80	0.45
			125	2.10	2.72	0.44
			210	2.32	2.60	0.42
			360	2.56	2.50	0.40
2	0.218	20	15	1.18	5.10	0.71
			30	1.48	4.92	0.69
			45	1.65	4.75	0.68
			60	1.78	4.67	0.67
			150	2.18	4.25	0.63
			270	2.43	3.94	0.60
3	0.238	25	20	1.30	9.4	0.97
			30	1.48	8.7	0.94
			60	1.78	8.0	0.90
			90	1.95	7.3	0.86
			120	2.08	7.2	0.86
			150	2.18	6.4	0.81
			330	2.52	6.3	0.80
			450	2.65	6.1	0.79
4	0.496	30	15	1.18	23.0	1.36
			30	1.48	21.8	1.34
			45	1.65	20.5	1.31
			60	1.78	20.0	1.30
			90	1.95	18.9	1.28
			120	2.08	18.2	1.26
			180	2.26	17.0	1.20
			330	2.52	15.5	1.19
4	0.496	33⅓	15	1.18	47.0	1.67
			30	1.48	39.0	1.59
			45	1.65	40.5	1.61
			75	1.88	35.5	1.55
			105	2.02	32.6	1.51
			180	2.26	31.2	1.50
			255	2.41	27.5	1.44
			330	2.52	26.2	1.42

The underlying cause of this change of viscosity with ageing is probably syneresis, which, although discussed in connection with bread by Ostwald (1919), Whymper (1922) and Verschaffelt and Van Teutem (1915), does not appear to have been described

as occurring in flour suspensions; although in the latter the change is probably one chiefly affecting the gluten, and, in particular, the jelly precursors already described; while in the staling of bread it appears that the starch is principally responsible. In a private communication, Hatschek, with whom these anomalies had been discussed, and who agreed that they were probably of syneretic origin, made the suggestion that it would be of interest to examine the behaviour of washed out gluten in this respect, and it was found on investigation that the phenomenon was strongly exhibited, carefully washed and kneaded gluten balls exuding a considerable amount of liquid if kept in a closed vessel over night; behaving in this respect in a manner very similar to weak gelatin gels.

TABLE V

VARIATION OF VISCOSITY FALL-BACK SLOPE WITH FLOUR CONCENTRATION

Concentration	Slope of fall-back curve
%	
33⅓	1.8
30	1.6
25	1.2
20	1.0
15	0.6
10	0.2

If citric acid is added to the water used in making up the dough before washing out the gluten, it is found that the syneresis is inhibited. A normal gluten ball containing 67.8% moisture after standing 24 hours, exuded 1.80 gm. syneresis liquid, whereas a gluten treated with citric acid containing 61.6% moisture exuded only 0.50 gm. liquid after standing for the same period. The same weight (10 gm.) of wet gluten was used in both cases.

In favour of the syneresis theory of fall back, it is noteworthy that a flour suspension, when brought by citric acid from its natural pH of 5.90 to pH 5.10, suffered a diminution in fall-back slope from 1.8 units to 0.4 units. Unless the experiment is carried out at a suitable pH, most irregular results are obtained and fall-back curves are given which are not rectilinear but S-shaped.

The effect of citric acid on syneresis of gluten and on fall-back slope is thus shown to be similar under suitable experimental conditions. The use of hydrochloric and sulphuric acids in place of citric acid at the same pH produced S-shaped curves for which a slope figure could not be obtained.

It has been suggested by Robertson (1918) that there may be a regular structure in protein suspensions analogous to the struc-

tures in ordinary ionic solutions proposed by Sutherland (1907), but since the fall back does not depend on shearing, which would tend to deform or partially destroy such a structure, the theory is scarcely applicable to flour suspensions, in which, moreover, the majority of the protein is by no means in a truly sol condition.

There is a slight change in pH in flour suspensions on standing, and though it is known that alteration of pH materially affects viscosity (giving an S-shaped curve with the isoelectric point at the minimum), the slight natural change of the order of 0.02 pH occurring during the measurement of a fall back does not appear to have any effect on the phenomenon, or produce any deviation from the straight line curve. The marked changes of viscosity caused by deliberate alteration of the pH appear to involve also the valency of the anion.

Section 3. Viscosity Temperature Coefficients

Chick and Martin (1912) have shown that both the viscosity–temperatures coefficients, and also the viscosity–concentration variations of protein suspensions are indications of the colloidal nature of the proteins, while the importance of the viscosity–concentration curve of flour suspensions as a measure of flour strength has been emphasized by Sharp and Gortner (1923), and is fully recognised by the writers (1927). Work has now been carried out on the correlation of the three variables, viscosity, temperature, and concentration, by plotting the slope of the time of fall-log temperature curves (which are rectilinear) against concentration, and it has been found possible to obtain an expression relating these for any given flour.

The method of Chick and Martin of plotting the concentration against viscosity, taking that of the dispersion medium as unity and of plotting the temperature against viscosities relative to water at the same temperature, has not been followed, in order to include all three factors in one expression. Although it might seem preferable to have expressed these results in absolute viscosity units, it would have been necessary in order to do so, to standardise a viscometer for both the concentration and the temperature variables, and a solid model or large series of curves would have been required. Since one viscometer is used throughout the whole of the temperature coefficient work, the error involved within this narrow range in considering the time of fall as a measure of the viscosity, is not great enough to justify such a large amount of work being done for a standardisation.

The straight line obtained as described above by plotting logarithmic temperature against time of fall follows the equation.

(5) .... $t = K \log \theta + b$

where θ is temperature

K and b are constants for any given concentration, and if we subtract from values of K a value of K_w where K_w is the value of K for water, it is found that plotting log $(K - K_w)$ against concentration, a straight line is also obtained. This straight line has the equation (6) ... $\log (K - K_w) = K' C + d$

Combining equations (5) and (6) we get

(7) $$\log \left[\frac{t - b}{\log \theta} - \frac{t_w - b_w}{\log \theta_w} \right] = K' C + d$$

which is the fundamental equation, where θ_w, t_w, and b_w refer to water, and are parameters.

The constants b and d are functions of the actual viscosity of the flour used.

Values of K′ were estimated in the case of three distinct flours, A, B, and C, of which it was known that A was of poor quality, B of average strength, and C strong.

The values of K′ found are almost identical, so that it does not appear that K′ is a function of strength. The full figures for these experiments are shown in Tables VI and VII, and the curves from which values of K were obtained are numbered 4 and 5. Curve 5 represents equation 7.

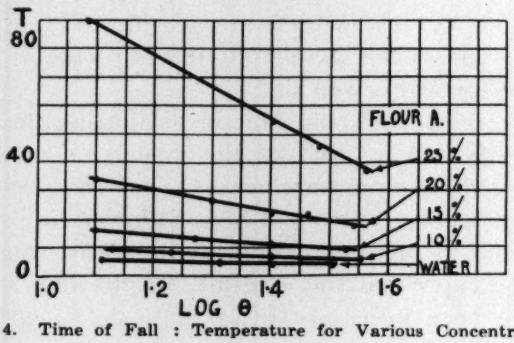

Fig. 4. Time of Fall : Temperature for Various Concentrations

It thus appears that the slope constant of the fundamental equation is approximately the same in the case of flours of widely different colloidal properties; but further work on this is in progress.

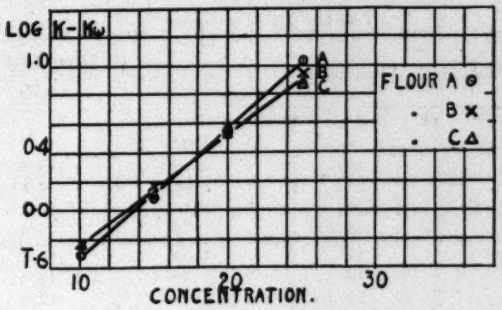

Fig. 5. Temperature Coefficient : Concentration

TABLE VI

VISCOSITY TEMPERATURE COEFFICIENT OF FLOUR SUSPENSIONS

(Viscometer bore 0.238)

Concentration	Temperature	Log temp.	Time of fall, mean of five readings
	Flour A		
%	°C.		sec.
10	13.5	1.13	9.6
	17	1.23	9.0
	25	1.40	7.9
	35.5	1.55	6.7
15	12.5	1.10	16.3
	18.5	1.27	14.1
	25	1.40	12.3
	33.5	1.53	10.5
20	12.5	1.10	34.5
	20	1.30	27.6
	25	1.40	22.7
	28.5	1.46	22.9
	34.75	1.54	18.5
25	12	1.08	90.5
	25	1.40	55.6
	30.5	1.48	46.0
	36	1.56	38.0
	Flour B		
10	14.25	1.15	9.8
	20	1.30	8.6
	24	1.39	7.8
	30	1.48	7.2
	35.5	1.55	6.7
15	10	1.00	18.4
	17.75	1.25	15.2
	24.25	1.39	12.6
	28.25	1.46	11.7
	34	1.53	9.9
20	11	1.04	36.4
	19.5	1.29	25.6
	24.75	1.39	21.5
	30.5	1.48	19.1
	34.5	1.54	17.9
25	10.25	1.01	83.5
	18.25	1.27	58.7
	23.5	1.37	47.9
	29.25	1.47	40.2
	36	1.56	34.3

TABLE VI—*Continued*

Concentration	Temperature	Log temp.	Time of fall, mean of five readings
	Flour C		
10	10.5	1.02	10.7
	15	1.18	9.6
	21	1.32	8.6
	30	1.48	7.2
	38.5	1.59	6.3
15	11	1.04	18.2
	19.5	1.29	14.4
	24.5	1.39	12.8
	30	1.48	11.4
	36	1.56	10.6
20	10.5	1.02	36.6
	15	1.18	29.7
	18.5	1.27	26.2
	25	1.40	22.8
	36	1.56	18.1
25	10	1.00	77.0
	18	1.26	59.3
	21	1.32	52.0
	25	1.40	46.7
	36	1.57	35.3
Water	13	1.11	5.75
	20.5	1.31	5.35
	25	1.40	5.2
	31	1.50	5.0

TABLE VII

VARIATION OF VISCOSITY TEMPERATURE COEFFICIENTS WITH CONCENTRATION FOR FLOUR SUSPENSIONS

Flour	Viscosity-temperature coefficient K	$K - K_w$	$\log (K - K_w)$	Concentration
				%
A	10.9	10.7	1.029	25
	3.6	3.4	0.532	20
	1.4	1.2	0.079	15
	0.7	0.5	$\bar{1}.699$	10
B	9.0	8.8	0.945	25
	3.6	3.4	0.532	20
	1.7	1.5	0.176	15
	0.8	0.6	$\bar{1}.778$	10
C	7.7	7.5	0.875	25
	3.95	3.75	0.574	20
	1.5	1.3	0.114	15
	0.8	0.6	$\bar{1}.778$	10

Summary

The Ostwald type of viscometer has been found entirely satisfactory for measurements on flour suspensions of concentrations up to 30%, if the instrument is calibrated with solutions of known viscosity, and the measurements are expressed in absolute

units. The dimensions of the capillary may be varied within wide limits without affecting the accuracy of the results, provided that the flow is laminar within the range of measurement and the critical velocity is not exceeded. The tendency of the viscosity of flour suspensions to decrease on standing (which was one of the objections raised by previous workers to this type of instrument) has been shown to be a definite colloidal phenomenon, which is not due to either repeated shearing or proteolysis, but probably to syneresis occurring in the suspensoid. A figure may be obtained for this viscosity fall back for any given flour, which is to some extent correlated with the strength of the flour.

The addition of citric acid produces a change in the slope of the fall back curve, and a similar change is produced in the syneresis of washed-out gluten to which citric acid has been added; other anions at the same pH, and citric at higher pH, give S-shaped instead of straight line curves.

The change of viscosity of flour suspensions with temperature has been examined, and an empirical equation relating the temperature coefficients with the concentration has been put forward.

It is found that the slope of the curve derived from this equation is independent of the colloidal properties of the flour used.

The authors wish to acknowledge the valuable assistance given by J. W. Dinsdale, who was responsible for the design of the viscometers and the early part of this work; and the stimulating suggestions and theoretical assistance of R. S. Willows, and Emil Hatschek.

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THE PREPARATION AND ANALYSIS OF THE VARIOUS PROTEINS OF WHEAT FLOUR WITH SPECIAL REFERENCE TO THE GLOBULIN, ALBUMIN, AND PROTEOSE FRACTIONS[1]

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Osborne (1907) has reported extensive studies on the proteins of wheat flour and records the isolation of five distinct proteins which differ from each other in composition, solubility, and other physical properties. These proteins are (1) a prolamine, gliadin; (2) a glutelin, glutenin; (3) an albumin, leucosin; (4) a globulin; and (5) a proteose.

Osborne reports that the albumin or leucosin forms 0.3 to 0.4 per cent of the wheat kernel. It is soluble in water, is coagulated by heating its solution, and is precipitated from solution by half saturation with ammonium sulfate. It resembles the animal proteins in elementary composition, in physical properties, and in amino acid content more closely than it resembles most of the proteins of cereals which have been studied. No analysis of this protein has been made except the elementary analysis and its content of several amino acids as determined by the Fischer ester method.

Osborne found the wheat kernel to contain about 0.6 per cent of a globulin which is insoluble in water but soluble in dilute saline solution. It is precipitated from its solution by saturation with magnesium sulfate. No analysis for this protein has been reported except its elementary analysis.

Osborne likewise found that the wheat kernel yields a very small amount of proteose. He did not isolate enough to study its amino acid content.

Since the publication of Osborne's work, extensive studies have been carried out on both gliadin and glutenin, but relatively few observers have reported data bearing on the composition or properties of the other three proteins. Recently Jones and Gersdorff (1923) have undertaken an investigation of the proteins of wheat bran and record the isolation of an albumin, a globulin, and a proteose which appear to differ from similar proteins isolated

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from wheat embryo by Osborne and Campbell (1900) and from the wheat kernel by Osborne and Voorhees (1893, 1894).

So little is known concerning the chemical composition and the properties of certain of these wheat flour proteins, that we have attempted to reinvestigate the question and to isolate a quantity in order that a comparison may be made of the chemical and physical properties of these proteins isolated from a single sample of wheat flour.

Circumstances compelled an alteration of plans before they were completely carried out so this study is not complete. It is thought, however, that the results obtained may be worthy of record.

Experimental

A patent flour of good baking quality, milled at the Minnesota State Experimental Mill, was used. The usual methods were employed for the isolation of glutenin and gliadin; and a somewhat modified procedure for isolating the albumin, globulin, and proteose. These are given in detail.

Gliadin.—Five kilos of flour was doughed up, allowed to stand under water, and the starch removed from the gluten by washing with tap water in the usual manner. The gluten was then cut into small pieces with a food chopper, and the fragments were extracted several times with 70 per cent alcohol at room temperature. The alcoholic solutions were united, filtered clear, concentrated to a syrup at low temperature under reduced pressure, and the protein was precipitated by pouring the concentrated alcoholic solution into water. The crude gliadin was redissolved in 70 per cent alcohol, filtered, and reprecipitated. The final purification was made by pouring the clear alcoholic solution into a mixture of absolute alcohol and ether. The precipitated gliadin was finally washed with ether and dried. A snow-white product was obtained. This sample contained 17.54 per cent nitrogen and 0.72 per cent sulfur on the ash- and moisture-free basis. Osborne reports 17.66 per cent nitrogen and 1.03 per cent sulfur.

Glutenin.—After the crude gluten had been completely extracted with 70 per cent alcohol, the residue was dispersed in a 0.5 per cent solution of sodium hydroxide. The alkaline solution was clarified by means of a Sharples supercentrifuge until practically water clear, and the protein was precipitated by the addition of acid until a pH of 4.5 to 5.0 was attained in the solution. The glutenin separated out, leaving a clear filtrate. The precipitate

was concentrated by means of the supercentrifuge. The crude product was purified by redissolving in 0.5 per cent sodium hydroxide and reprecipitating. A final purification was effected by dissolving the protein in a small amount of 0.5 per cent sodium hydroxide and adding this to about four volumes of methyl alcohol, filtering, and precipitating the glutenin by adding dilute hydrochloric acid, as described by Blish and Sandstedt (1925). The precipitated protein was then thoroly extracted with 70 per cent alcohol to remove any final traces of gliadin and was dried by washing with absolute alcohol followed by a mixture of absolute alcohol and anhydrous ether. A pure white powder resulted which contained (ash- and moisture-free) 17.20 per cent nitrogen and 0.96 per cent sulfur. Osborne reports 17.49 per cent nitrogen and 1.08 per cent sulfur.

Globulin.—Eighteen kilos of the flour were extracted with 5 per cent potassium sulfate solution in the ratio of 1 kilo of flour to 3 liters of the salt solution. This was accomplished by adding the flour to the salt solution during vigorous agitation, so that a fine suspension resulted. After several hours the flour had settled so that the fairly clear supernatant liquid could be siphoned off. As a rule the volume of the extract obtained amounted to about 70 per cent of the volume of the potassium sulfate solution which had been added. The combined extracts were completely clarified by passing through a supercentrifuge. The clarified solutions were then dialyzed in large "Cellophane" bags against running water until nearly free of sulfates. The protein which separated was removed by again passing the mixture through the supercentrifuge, reserving the clarified liquid for the preparation of leucosin.

The protein was removed from the centrifuge bowl, and attempts were made to purify it by re-solution in 5 per cent potassium sulfate solution. Unfortunately, it proved to be practically insoluble in either 5 per cent potassium sulfate or 10 per cent sodium chloride solutions, only traces dissolving after standing several days in contact with the salt solution.

The portion insoluble in 5 per cent potassium sulfate was dissolved in a small amount of 0.2 per cent sodium hydroxide. This alkaline solution was filtered and neutralized with hydrochloric acid. A heavy, easily filterable precipitate was obtained. This was filtered off, washed several times with 70 per cent alcohol, then with absolute alcohol, and finally with anhydrous ether. The yield was 5.8 grams of a light gray powder. This was designated

as "globulin A." The ash- and moisture-free sample contained only 14.77 per cent nitrogen.

Because this preparation of protein was not soluble in either 5 per cent potassium sulfate or 10 per cent sodium chloride, it was thought that perhaps we were not dealing with a globulin; accordingly another preparation was attempted, using 10 per cent sodium chloride solution as originally employed by Osborne for the preparation of the globulin fraction.

Fourteen kilos of flour were extracted with 10 per cent sodium chloride solution in the ratio of one kilo of flour to 6 liters of the solution. A more concentrated extract could not be obtained, owing to the striking difference in behavior between the 5 per cent potassium sulfate extracts and the 10 per cent sodium chloride extracts. The potassium sulfate extracts were of low viscosity, and the flour particles settled readily. The 10 per cent sodium chloride extracts were very viscous and because of the high viscosity, the flour particles settled slowly. Even with a ratio of one kilo of flour to 6 liters of salt solution it was impossible to siphon off more than 50 per cent of the liquid originally added. This liquid was filtered, clarified in the supercentrifuge, and dialyzed in Cellophane bags against running water, to a low chloride content. The protein which separated was filtered off, and the filtrate was reserved for the preparation of the albumin. The crude globulin was redispersed as completely as possible in 10 per cent sodium chloride; the solution was filtered and again dialyzed in Cellophane bags until free of chlorides. The precipitated protein was washed first with 70 per cent alcohol, then with absolute alcohol, followed by ether. The yield was 3.1 grams air-dry with a nitrogen content on the ash- and moisture-free basis of 18.26 per cent. This product was designated as "globulin B."

This preparation differed from globulin A in that the crude product was nearly all soluble in 10 per cent sodium chloride solution. There is a possibility that in the first preparation the initial dialysis was carried too far, so that no salt remained in contact with the protein, thus causing a protein⟶protean transformation. However, other suggestions, which will be reserved for discussion in a subsequent paper, may explain the difference in properties.

Albumin (Leucosin).—After the globulins had been removed from the 5 per cent potassium sulfate extract, as noted above, the filtrates were concentrated *in vacuo* at a temperature below 50° C. to a volume of about 4 liters and filtered clear. The albumin

was precipitated from this solution by half saturation with ammonium sulfate. The precipitated albumin was removed from the solution by means of the supercentrifuge and the precipitate was washed in the supercentrifuge with half-saturated ammonium sulfate. The filtrate was reserved for the "proteose" preparation.

The crude albumin was dissolved in water, the solution filtered and half saturated with ammonium sulfate. The albumin separated very sharply, leaving a clear supernatant liquid. The albumin precipitate filtered very readily, having in this respect the characteristics of crystals. The precipitate was washed with half saturated ammonium sulfate, then redissolved in water and dialyzed in Cellophane bags until free of sulfates. The solution of albumin was perfectly clear but had a dark brown (or black in concentrated solution) color. After dialyzing, the albumin was not heat-coagulable. A trace of acid, however, would cause the albumin to coagulate by heat. The preparation gave a strong Millon's test. The albumin was precipitated by pouring the aqueous solution into alcohol. The precipitated albumin was filtered off, washed with alcohol and ether, and dried. The yield was 21 grams of a dark brown powder. This was designated as "albumin A." The preparation contained 16.73 per cent nitrogen and 3.42 per cent sulfur. It is possible that the high sulfur value may be due in part to traces of adsorbed ammonium sulfate or combined sulfuric acid which it was impossible to remove by the dialysis procedure used.

A second preparation of albumin was made from the filtrate from the globulin of the 10 per cent sodium chloride extract. For the preparation of the albumin these filtrates were treated exactly as were those from the potassium sulfate extracts. Rather surprisingly, the yield of albumin was extremely small, approximately 1.0 grams. The nitrogen content on the air-dry basis was only 13.51 per cent.

Proteose.—Osborne prepared a proteose from the filtrates from the albumin precipitate by completely saturating them with ammonium sulfate. Accordingly, solid ammonium sulfate was added to the filtrate from the albumin A precipitate until complete saturation was obtained. A voluminous precipitate separated. This product was separated by passing the mixture through the supercentrifuge. The crude product was redissolved in water, and the solution was filtered. The aqueous solution was extremely viscous. Several hours standing in contact with about a liter of

water was required before complete solution was attained. When the solid material was added to water it became strongly hydrated and its solution process resembled that of gum arabic.

The clear solution was then exactly half saturated with ammonium sulfate in order to precipitate any albumin which might still contaminate the solution. No turbidity resulted. A considerable amount of ammonium sulfate could still be added before a precipitate formed and finally, at practically complete saturation with ammonium sulfate, a heavy flocculent precipitate separated very sharply. This precipitate, which caked together in a mass, was drained as free as possible from the mother liquor and after redissolving in water was dialyzed in Cellophane bags to the entire absence of tests for the sulfate ion. A clear, slightly yellowish solution resulted. This solution was dialyzed against 95 per cent alcohol which precipitated a light gray powder. This was filtered off, washed with 95 per cent alcohol, absolute alcohol, and ether, and dried. The product weighed 29 grams and was light gray in color. It did not give Millon's reaction and to our surprise *was found on analysis to contain no nitrogen.* A solution of this product, when tested with an iodine solution, did not become blue, consequently it contained no starch. It did not reduce Fehling's solution until after it had been hydrolyzed by heating with acids, when abundant reduction was obtained. It appears to be a hitherto undescribed gum which is present in the wheat flour. A detailed chemical study of this product is reserved for a later paper. Suffice it to say here that its solutions are among the most viscous that it has been our privilege to encounter. No trace of proteose was isolated from the 5 per cent potassium sulfate extracts.

The corresponding filtrates from the albumin fraction of the 10 per cent sodium chloride extractions were worked up similarly. No trace of proteose was obtained. Here again the "gum" was isolated but the yield was only 3.10 grams.

Van Slyke analyses of protein isolated.—The various proteins were subjected to an analysis, using the method suggested by Van Slyke (1911). The data are shown in Table I. The analyses for gliadin, glutenin, and albumin A, and globulin A represent average values of duplicate analyses. Only 0.7180 gram (97.27 mgr. N) of albumin B and 1.6628 grams (343.2 mgr. N) of globulin B were available for analyses, so these columns represent single determinations. Obviously the experimental error in the values for albumin B is large.

TABLE I

NITROGEN DISTRIBUTION (IN PER CENT OF TOTAL NITROGEN) BY THE VAN SLYKE METHOD OF THE VARIOUS PROTEINS SEPARATED FROM A SINGLE SAMPLE OF WHEAT FLOUR

	Gliadin	Glutenin	Albumin A	Albumin B*	Globulin A	Globulin B†
	%	%	%	%	%	%
Ammonia nitrogen	25.90	13.56	10.67	10.28	8.28	10.60
Insol. humin nitrogen	0.34	0.78	0.93	1.14	2.13	0.59
Soluble humin nitrogen	0.32	0.66	1.13	2.59	1.26	0.49
Arginine nitrogen	5.29	11.96	15.70	10.69	14.29	18.64
Histidine nitrogen	8.22	4.72	4.44	4.63	1.29	11.27
Lysine nitrogen	0.50	4.52	9.50	4.81	6.84	2.39
Cystine nitrogen	0.44	0.67	1.63	1.87	2.39	2.04
Amino nitrogen in filtrate	53.23	53.47	49.47	50.84	55.91	42.42
Non-amino nitrogen in filtrate	5.95	7.50	7.04	17.01	7.87	11.42
Total	100.19	97.84	100.51	103.86	100.26	99.86
Total nitrogen in protein	17.54	17.20	16.73	13.51‡	14.77	18.26
Total sulfur in protein	0.76	0.96	3.42	....	....	....

* A single determination on 0.7180 grams on air-dry basis.
† A single determination on 1.6628 grams ash- and moisture-free protein.
‡ Air-dry basis. Moisture and ash determinations were not run on this sample.

The analysis of gliadin agrees fairly well with those recorded in the literature (cf. Bailey, 1925) except that the histidine value is somewhat high and the non-amino nitrogen in the filtrate from the bases is correspondingly low.

The ammonia nitrogen of the glutenin is lower than any reported by Blish (1916), but is not quite so low as some reported by Cross and Swain (1924). In general, the glutenin analysis agrees fairly well with values reported by Cross and Swain, altho our histidine value is lower than any of the recorded values.

Comparative analyses by other workers for the albumin and globulins of wheat flour are not available. Lüers and Landauer (1922) have reported Van Slyke analyses for an albumin which they call "leucosin" but which was isolated from finely milled barley by extraction with water.

It is our belief that the analysis of albumin A is essentially correct. The low ammonia nitrogen indicates that the dialysis for the removal of ammonium sulfate was highly satisfactory. The small amount of albumin B which was available probably introduced errors. Note in particular that the arginine value is low and the non-amino nitrogen in the filtrate is high, a result which would be brought about by incomplete separation of the arginine phosphotungstate. The nitrogen distribution of albumin A is very similar to the analyses reported by Lüers and Landauer for the albumin of barley, the only striking difference being in the nitrogen in the filtrate from the bases, where they report 60.44 per cent of amino nitrogen and 2.67 per cent of non-amino nitrogen.

The low total nitrogen content of globulin A casts doubts on the value of this analysis and the small amount of globulin B likewise places a question on these values. Consequently, neither of these analyses should be regarded as satisfactory, altho the ammonia values are probably fairly accurate, as ammonia nitrogen can be determined with a high degree of precision.

Certain experiences in making the separations reported in this paper, together with subsequent experience to be reported later, make us strongly doubt whether or not any appreciable amount of a true globulin exists in patent wheat flour. We are certain that if a globulin is present it is in extremely small amounts. We are equally certain that no appreciable amount of proteose was present in the sample of flour which we examined.

Summary

1. A sample of patent wheat flour has been so treated as to separate the various proteins. Preparations having the properties of a prolamine (gliadin), a glutelin (glutenin), and an albumin (leucosin) were obtained. Small amounts of a possible globulin were likewise isolated. No proteose could be detected.

2. The extraction of wheat flour with 5 per cent potassium sulfate solution and 10 per cent sodium chloride solution does not yield similar fractions. Marked differences were observed in the substances dissolved by these solutions and in the amount of proteins which could be separated from such solutions.

3. The nitrogen distribution by the Van Slyke method has been determined for the various proteins isolated.

4. The sample of wheat flour examined contained a gum which was isolated in, apparently, a pure condition. This gum did not color blue with iodine, nor did it reduce Fehling's solution before it was subjected to acid hydrolysis.

5. The authors believe that the presence of a true globulin in certain samples of wheat flour is problematical.

6. No evidence was obtained that would suggest the presence of a proteose in this sample of wheat flour.

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THE CONCENTRATION OF GLUTENIN AND OTHER PROTEINS IN VARIOUS TYPES OF WHEAT FLOUR[1]

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In 1896 the French chemist Fleurent (1896a. 1896b) suggested that the ratio of gliadin to glutenin in wheat flour was correlated with baking strength. He prepared crude gluten from flour by washing a simple flour-water dough with water. This crude gluten was dispersed in a 70% solution of alcohol containing 0.3% of potassium hydroxide. The glutenin in this preparation was precipitated by bubbling carbon dioxide through the alcoholic hydroxide solution. The gliadin was then removed by filtering the solution and drying an aliquot of the solution. The fraction of the crude gluten represented by the gliadin was then computed and the non-gliadin portion of the gluten was considered to be glutenin. In computing the percentage of gliadin, a correction was made for the potassium carbonate. Fleurent concluded that gluten of good quality was composed of approximately 75% of gliadin and 25% of glutenin when the two proteins were determined by his method.

Snyder (1899a, 1899b, 1901) also studied the correlation of the gliadin-glutenin ratio with flour strength. The method he used was somewhat different from that employed by Fleurent. The gliadin in flour was determined by a direct extraction with 70% alcohol, and the nitrogen in the protein of this alcoholic extract was then determined by the Kjeldahl method. The flour residue from the alcohol extraction was treated with 5% sodium chloride solution to remove the salt-soluble proteins. The nitrogen content of the residue was then determined and recorded as glutenin nitrogen. Snyder concluded that the optimum ratio was 65% of gliadin to 35% of glutenin in flours of superior baking strength.

Guess (1900) and Norton (1906) contended that the gliadin-glutenin ratio might be regarded as a quality factor and that such a factor multiplied by the percentage of gluten would

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afford a measure of baking value. Kosutany (1903, 1909) supported the contention that the ratio of gliadin to glutenin is of importance. Martin (1920) "amended" the gliadin figure by deducting the percentage of protein extracted from flour by water from the percentage of alcohol-soluble protein. Such amended gliadin values were found to be high in the flours with superior gas-retaining capacity and bakers' marks which were examined by Martin.

Certain other chemists seemingly found little of significance in the ratio of gliadin to glutenin or in the gliadin content of flour. König and Rintelen (1904), Wood (1907), Shutt (1910), Rammstedt (1909), and others concluded that the concentration of these proteins was not a determining factor in the strength of flour. This failure to agree with the conclusions reached by Fleurent, Snyder, and Martin may probably be attributed in part to the difficulties which existed in accurately determining the percentages of these proteins in flour.

In the data recorded by Blish (1916), Sharp and Gortner (1923), and Blish and Sandstedt (1925), and in the computations made by Zinn (1923) there appears to be a basis for assuming that the ratio of gliadin to glutenin in the ordinary types of flour may be relatively constant. It was deemed advisable, however, to extend the studies that had been made to cover representative samples of all types of flour, and to use methods recently developed in an effort to ascertain the extent of variation in the several protein fractions of these flours.

Experimental

Source and description of flour samples.—Seventeen flour samples were collected from various parts of the United States and Canada. They were either patent or straight grade flours and were milled from soft western wheat, soft red winter wheat, hard winter wheat, and hard spring wheat. The description of these samples as given by the millers is as follows:

No. 1. Straight Pacific Coast soft wheat flour milled from a mixture of Club, and soft white wheat. Flour bleached lightly by both the Alsop and the chlorine processes. It required 260 pounds of cleaned wheat to produce one barrel of 196 pounds of straight grade flour.

No. 2. First patent flour, representing 55 per cent of the total flour milled from Canadian spring wheat. Flour bleached by the Alsop process.

No. 3. An 80 per cent patent milled from hard spring wheat yielding 70 per cent of total flour. Flour bleached by the Alsop and chlorine processes.

No. 4. An 80 per cent patent milled from hard winter wheat yielding 71 per cent of total flour. Sample bleached by the Alsop and chlorine processes.

No. 5. A 50 per cent patent milled from Canadian hard spring wheat yielding 72 per cent of total flour. Sample bleached by the Alsop process.

No. 6. An 82 per cent patent milled from a mixture of Turkey Red and Kanred (hard red winter) wheat requiring 4 bushels and 27 pounds of wheat to produce one barrel of 196 pounds of flour.

No. 7. A patent flour representing 48 per cent of the total flour milled from a blend of soft winter wheat grown in Indiana and Ohio. The total yield of flour from the wheat was 72.2 per cent.

No. 8. Short patent soft winter wheat flour milled in Indiana.

No. 9. Straight grade hard spring wheat flour milled at Minnesota Experimental Flour Mill, their serial No. 206. Weight per bushel of wheat, 60 pounds; yield of flour, 75.72 per cent.

No. 10. Straight grade hard spring wheat flour from same source as No. 9, their serial No. 214. Weight per bushel of wheat, 59.8 pounds; yield of flour, 72.54 per cent.

No. 11. Straight grade hard spring wheat flour from same source as No. 9, their serial No. 210. Weight per bushel of wheat, 60.5 pounds; yield of flour, 73.27 per cent.

No. 12. Straight grade hard spring wheat flour from same source as No. 9, their serial No. 216. Weight per bushel of wheat, 60.7 pounds; yield of flour, 71.57 per cent.

No. 13. A 95 per cent standard patent milled from Ohio red winter wheat. Flour bleached by the Alsop process.

No. 14. Patent flour milled from a blend of hard winter wheat grown in western Nebraska and hard spring wheat, the mixture including 1.5 per cent of malted wheat.

No. 15. A 40 per cent patent milled from soft red winter wheat, chiefly from Missouri and largely of Fulcaster and Fultz varieties. In milling this wheat, 4 bushels and 39 pounds were required to produce a barrel (196 pounds) of flour. Flour bleached by the Agene process with nitrogen trichloride.

No. 16. Straight grade flour milled from No. 1 hard winter wheat grown in Montana. Wheat was ground at the Minnesota

State Experimental Flour Mill and yielded 76.3 per cent of straight grade flour.

No. 17. Straight grade flour milled from durum wheat.

Methods Used in Protein Separations

These flours were first subjected to chemical analysis in an effort to estimate the concentration of the several proteins in the samples. Total protein was determined by the Kjeldahl-Gunning method. In the determination of the proteins soluble in 5% potassium sulfate solution, a departure was made from the tentative A.O.A.C. method. A charge of 6 grams of flour was placed in a 100-cc. centrifuge tube and 75 cc. of the sulfate solution was added, with a drop of thymol solution to inhibit bacterial activity. The flour was suspended in the sulfate solution by vigorous agitation and the tube and contents were then shaken in a mechanical shaker for 30 minutes. The tube was then placed in a centrifuge which was rotated rapidly to precipitate the flour. The supernatant liquid was decanted into a Kjeldahl flask. The extraction process was repeated with two additional portions of 75 cc. of sulfate solution, the precipitated flour being suspended and shaken 30 minutes in each instance. The three extracts were combined in the same Kjeldahl flask and the percentage of nitrogen in the extract was determined by the Kjeldahl-Gunning method. The concentration of nitrogen, in percentage, multiplied by the factor 5.7 was recorded as "potassium sulfate-soluble nitrogen," which fraction consists in large part of albumin and globulin, as shown by the work of Bailey and Blish (1915).

In order to determine the effectivenes of this procedure, the percentage of protein in each of four successive extracts was determined on three of the samples. The resulting data in terms of protein are recorded in Table I. From these data it is evident that only traces of protein soluble in 5% potassium sulfate solution remain in the flour after the second extraction, and the third extraction probably took out everything that could be regarded as nongluten proteins.

Proteins soluble in 70% ethyl alcohol were determined by extracting the residue from the sulfate extraction with alcohol of that concentration. The flour particles in the residue were suspended by shaking, and were kept in suspension by moderate shaking in a mechanical shaking machine. The flour particles were then precipitated by whirling in the centrifuge and the supernatant

liquid was decanted into a Kjeldahl flask. The extraction was repeated twice with 70% alcohol, the three extracts being combined in the same flask. The nitrogen in the alcoholic extract was determined by the Kjeldahl-Gunning method and the percentage of nitrogen multiplied by the factor 5.7 was recorded as gliadin or alcohol-soluble protein.

TABLE I

PROTEINS EXTRACTED FROM 3 SAMPLES OF FLOUR WITH FOUR SUCCESSIVE PORTIONS OF 5% POTASSIUM SULFATE SOLUTION

Extraction No.	Sample No. 15	Sample No. 16	Sample No. 17
	(N x 5.7)	(N x 5.7)	(N x 5.7)
	%	%	%
1	1.32	1.79	2.02
1	1.30	1.79	1.96
2	0.24	0.34	0.39
2	0.24	0.32	0.36
3	0.10	0.13	0.15
3	0.09	0.09	0.15
4	0.08	0.08	0.07
4	0.09	0.13	0.08

Glutenin. Method I.—The insoluble residue remaining after the extractions with aqueous 5% potassium sulfate solution and 70% ethyl alcohol solution was transferred to a Kjeldahl flask and the nitrogen content determined by the Kjeldahl-Gunning method. The percentage of nitrogen multiplied by the factor 5.7 was recorded in Table II as "Glutenin by Sharp and Gortner Method I."

Glutenin. Method II.—The glutenin content of the 17 flours was also determined by the direct method of Blish and Sandstedt (1925). This method involves the dispersion of the proteins in sodium hydroxide solution, the addition of methyl alcohol, and the neutralization of the alkali with hydrochloric acid. Glutenin precipitated on neutralization is collected by whirling in a centrifuge tube and the determination of the nitrogen in the precipitate. The percentage of nitrogen multiplied by the factor 5.7 is recorded as "Glutenin by the Blish and Sandstedt Method II" in Table II.

It will be observed that the two methods gave similar results in all but three of the samples, Nos. 5, 16, and 17. In the other instances the results by the two methods were practically within the limits of the experimental error of a single method.

Ratios of Protein Fractions

Three series of calculations were based upon these protein determinations. The first involved the calculation of the ratio of the protein extracted by 5% potassium sulfate solution to the crude protein, or B/A. This ratio varied from 0.157 to 0.223. The

TABLE II

COMPOSITION OF THE 17 FLOURS (DRY BASIS) AND PROTEIN RATIOS

Lab. No.	Ash	Crude protein (N x 5.7) (preparation A)	Protein extracted in 5% K_2SO_4 solution (preparation B)	B / A	"Gliadin" or protein extracted with 70% alcohol (preparation C)	Glutenin by Sharp and Gortner method I (preparation D)	Glutenin by Blish and Sandstedt method II	D / A	D / C+D
	%	%	%		%	%	%		
1	0.62	9.73	2.16	0.22	4.38	3.50	3.48	0.36	0.44
2	.51	13.99	2.40	.17	6.30	5.20	5.31	.37	.45
3	.48	13.07	2.21	.17	5.47	5.20	5.02	.40	.49
4	.45	12.53	2.00	.16	5.52	4.85	4.85	.39	.47
5	.47	14.29	2.45	.17	6.79	5.02	5.62	.35	.45
6	.51	12.34	2.19	.18	5.35	4.58	4.51	.37	.46
7	.43	10.61	1.80	.17	4.82	3.98	3.96	.37	.45
8	.51	10.63	2.05	.19	4.54	3.95	4.04	.37	.46
9	.58	12.99	2.35	.18	5.30	5.17	5.13	.40	.49
10	.57	14.10	2.50	.18	5.81	5.67	5.50	.40	.49
11	.58	11.56	2.18	.19	5.01	4.40	4.50	.38	.47
12	.62	12.24	2.26	.18	5.44	4.60	4.78	.37	.46
13	.46	10.09	2.08	.21	4.32	3.67	3.64	.36	.46
14	.52	12.76	2.53	.20	5.63	4.74	4.58	.37	.46
15	.42	12.49	1.96	.16	5.35	4.49	4.35	.38	.46
16	.49	14.45	2.34	.16	7.07	4.94	5.52	.34	.41
17	0.86	13.64	2.55	0.18	6.47	4.56	5.53	0.33	0.41

highest ratio is encountered in Pacific Coast soft wheat flour, altho this flour did not contain the highest percentage of protein extracted by the sulfate solution. While the highest percentage of protein was extracted from the durum flour with the 5% potassium sulfate solution, the ratio of this fraction to the crude protein in this flour was no greater than that in certain spring wheat flours (Nos. 11, 12, and 13), which were stronger flours as judged by the baking tests recorded in Table III.

The second computation involved the ratio of glutenin (method I) to crude protein (D/A) in each of the 17 flour samples. The resulting data are recorded in the ninth column, Table II. In general, there is no substantial variation in this ratio in these flours of widely varying characteristics. The coefficient of correlation of glutenin and total protein content was 0.901 ± 0.031. The lowest ratio of glutenin to crude protein, 0.33, was in Sample 17, milled from durum wheat. The ratio in this sample is equivalent to about the lowest ratio observed by Vogel and Bailey (1927), who reported variations in the latter from 0.34 to 0.40 in 18 durum flour samples, with an average ratio of 0.37. The highest ratio of gliadin to crude protein in the 17 flours was 0.40 in three hard spring wheat flours—Nos. 3, 9, and 10. The range is not large and 14 of the 17 samples fell within the relatively narrow range of 0.36 to 0.40. Among these 14 samples were included as widely different types of flours as low-gluten Pacific Coast wheat flour, high-protein spring wheat flour, and intermediate degrees of protein content among the hard winter and soft winter wheat flours. It accordingly appears that the glutenin-protein ratio will not serve to distinguish between the various types of flour, since no regular or substantial variations in this characteristic were seen.

The third computation involved the ratio of glutenin (Method I) to the sum of gliadin and glutenin $\frac{D}{(C + D)}$. The data resulting from such calculations are recorded in the right hand column of Table II, and represent essentially the type of calculation formerly designated as the gliadin-glutenin ratio. It is evident that the percentage of glutenin in true gluten (glutenin + gliadin), as determined in these studies, is somewhat greater than the ratio previously reported by Fleurent and by Snyder. The range in this ratio in these 17 samples was from 0.41 to 0.49. Fifteen of the 17 samples fell within the relatively narrow range of 0.44 to 0.49. As in the glutenin-protein ratio, the differences are so small as to justify the conclusion that there is no substantial variation in the

proportion of gliadin and glutenin in flours of widely different types.

Baking Tests of the Flours

The 17 flours used in this study were subjected to baking tests following the method suggested by the Committee on Standard Formula and Method of Procedure for Experimental Baking Tests, of the American Association of Cereal Chemists (1925). The formula of the dough prepared from each flour was as follows:

Flour	325 grams
Sugar	10 "
Salt	5 "
Compressed yeast	10 "
Shortening	7 "
Water	sufficient

The absorption of each flour was first determined by doughing up 25 grams of flour, and noting the quantity of water required to produce a dough of a definite consistency. Before the final baking tests were conducted, several trial tests were made with each flour to establish further the correct absorption, as it is generally accepted that doughs may stiffen or slacken during fermentation. The absorption recorded in Table III appeared to give the best bread from each of the flours. The loaf volume and texture score of the loaves produced in the final baking trials are recorded in the same table. These data show substantial variations in baking quality of the flours used. The poorest bread was produced from Sample No. 1, milled from Pacific Coast soft wheat. The next appeared to be the durum wheat flour (Sample No. 17). Superior bread was made from the hard wheats, altho Flour No. 15, milled from Missouri soft red winter wheat, made very satisfactory bread.

TABLE III

RESULTS OF BAKING TESTS MADE AFTER THE PROCEDURE RECOMMENDED BY THE COMMITTEE OF THE AMERICAN ASSOCIATION OF CEREAL CHEMISTS (1925)

Lab. No.	Absorption	Loaf volume	Texture score
	%	cc.	
1	56.9	1810	90
2	68.8	1990	97
3	60.0	2180	97
4	64.0	2130	97
5	66.5	2170	99
6	60.0	2140	99
7	57.5	1990	97
8	56.0	1970	96
9	59.0	2040	97
10	59.5	2150	97
11	64.0	1980	97
12	62.5	2030	98
13	58.5	2020	97
14	57.5	2150	98
15	58.0	2200	99
16	58.5	2110	97
17	60.0	1940	95

Ten of the samples were baked by two additional methods, namely, the method used in the laboratory of the Minnesota State Experimental Flour Mill, and that used in the Department of Agricultural Chemistry, of the Nebraska Agricultural Experiment Station. The formulas are as follows:

Ingredients	Minnesota State Experimental Mill formula	Nebraska Experiment Station formula
Flour	350.0 grams	325 grams
Salt	5.25 "	7 "
Sugar	10.5 "	15 "
Yeast	10.5 "	15 "
Shortening	5.5 "	10 "
Water	sufficient	sufficient

Both methods involve fermenting the dough at 30° C. and proofing or raising the dough in the pans at 34° C.

The results of these baking tests are recorded in Table IV. Loaf volumes were recorded in terms of cubical displacement in cubic centimeters, and also in terms of the control or "standard" flour that was included in both series of baking tests. The latter computation affords a basis for determining the relative volume of loaves baked by the two methods. When baked by the Minnesota Mill method, the loaves were substantially larger than the standard or control when flours Nos. 2, 6, and 9 were used; and when baked by the Nebraska Station method they were smaller than the control or standard. The order of loaf volume was not the same in the two methods.

These differences and the results obtained in baking a series of flours by several methods, emphasize the necessity of carefully selecting a procedure for testing baking quality of commercial flours.

The coefficient of correlation of the percentage of crude protein in the 17 flours with the volume of loaf in cubic centimeters, as produced by the method of the American Association of Cereal Chemists, was 0.678 ± 0.091. This is a relatively large positive correlation, being greater than that previously reported by Zinn (1923), Mangels and Sanderson (1925), Blish and Sandstedt (1925), Mangels (1926), and Bailey and Sherwood (1926). The coefficient of correlation of loaf volume with glutenin content was 0.630 ± 0.099, or approximately the same as the correlation of loaf volume and protein content, as might be anticipated from the relatively constant ratio of glutenin to protein content. It is recognized that the number of samples involved in this computation is relatively small and chance may have contributed to the magnitude of the correlation coefficient. The same comment applies to the other calculations reported in this paper.

TABLE IV

RESULTS OF BAKING TESTS OF CERTAIN FLOUR SAMPLES, FOLLOWING THE REGULAR TESTING METHODS OF THE STATE MILL, AND THE METHOD USED BY THE NEBRASKA AGRICULTURAL EXPERIMENT STATION

Lab. No.	Absorption	Color score	Texture score	Loaf volume	
				Actual	In terms of the standard flour
	%			cc.	%
State Mill Method					
2	65.4	100	100	2380	118.2
3	61.7	98	100	2130	106.7
4	61.7	98	100	2050	101.9
5	63.4	100	100	2290	113.9
6	58.6	99	100	2210	109.9
9	60.0	97	99	2140	106.4
10	64.3	98	96	1900	94.5
11	60.0	97	98	2260	112.4
12	63.4	96	96	1870	93.3
14	60.6	99	100	2150	106.9
"Standard"	64.3	100	100	2010	100.0
Nebraska Experiment Station Method					
2	66.1	100	99	2140	99.5
3	65.2	100	99	2400	111.1
4	63.0	99	99	2260	105.1
5	68.3	99	99	2220	103.2
6	62.2	100	98	2120	98.6
9	60.1	97	99	1950	90.7
10	66.8	97	97	1870	86.9
11	69.2	97	98	2290	106.7
12	60.1	97	97	1900	88.4
14	60.1	100	99	2400	111.1
"Standard"	66.1	100	100	2150	100.0

Extensibility of doughs.—Chopin (1921) devised an instrument for quantitatively measuring the extension of surface when dough is stretched to the point where it breaks. The method used in testing the 17 samples in this series was essentially the same as that described by Bailey and LeVesconte (1924). A sample dough was prepared by mixing flour with 2% common salt solution in a mechanical dough mixer for 8 minutes, the quantity of salt solution being so adjusted as to give doughs of about the same degree of consistency. This dough was allowed to stand for 20 minutes, rolled into a sheet and cut into 10 biscuits, each of which was tested separately in the Chopin extensimeter. As two doughs were prepared in each instance, the determinations of extensibility were replicated twenty times. The averages of these 20 measurements are recorded in the last column of Table V. In this table is also recorded the loaf volume of bread produced by the method of the American Association of Cereal Chemists. These data show that flours which produced bread of the largest volume likewise gave the highest results when tested with the Chopin instrument

The parallelism between loaf volume and extensibility is not exact, but when the coefficient of correlation of these two variables was calculated, it was found to be 0.671 ± 0.076, which is approximately the same as the correlation between loaf volume and protein content.

TABLE V

COMPOSITION AND PROPERTIES OF THE FLOURS EXAMINED

Lab. No.	Moisture	Crude protein	Loaf volume	Ash	Quality constant b	(Glutenin). (b) / Loaf volume	Extensibility with Chopin device
	%	%	cc.	%			
1	9.08	8.85	1810	0.56	2.63	0.00508	15.7
2	10.13	12.57	1990	.46	2.49	.00650	25.2
3	10.31	11.72	2180	.43	2.51	.00598	23.3
4	12.53	10.97	2130	.40	2.90	.00654	22.6
5	11.13	12.71	2170	.42	2.50	.00578	23.8
6	11.83	10.87	2140	.45	2.90	.00620	22.8
7	11.86	9.35	1990	.38	2.74	.00509	19.3
8	12.16	9.34	1970	.44	2.26	.00453	20.3
9	12.15	11.42	2040	.51	2.17	.00549	23.8
10	11.71	12.45	2150	.50	2.28	.00602	25.9
11	11.36	10.25	1980	.51	2.39	.00531	20.8
12	11.71	10.81	2030	.55	2.77	.00582	24.7
13	10.49	9.04	2020	.41	2.16	.00393	19.7
14	11.87	11.25	2150	.46	2.54	.00560	23.1
15	9.93	10.67	2200	.38	2.34	.00482	21.8
16	9.90	13.02	2110	.44	2.30	.00538	26.8
17	9.81	12.30	1940	0.78	2.30	0.00538	17.1

Viscosity of flour suspensions.[2]—The viscosity of flour suspensions has been suggested as a criterion of the colloidal condition and properties of the glutenin of wheat flour. Early studies of viscosity of flour pastes were reported by Lüers and Ostwald (1919). Sharp and Gortner (1923) conducted an extensive study of viscosity of flour preparations and traced the effect of several variables upon the viscosity of acidulated suspensions. They contended that an acceptable expression of the colloidal condition of glutenin would be found in the angle which the curve forms with the abscissas when the logarithms of concentrations of flour are plotted as abscissas and the logarithms of viscosity of leached and acidulated flour suspensions are plotted as ordinates. Such a plot approaches the form of a straight line, and if the observed values deviate slightly from the theoretical straight line, the values are corrected to the theoretical by the method of least squares, using the following formula:

$$b = \frac{\Sigma (x).\ \Sigma (y) - n\ \Sigma (xy)}{[\Sigma (x)]^2 - n\ \Sigma (x^2)}$$

2 For more detailed consideration of the colloidal properties of flour as evidenced by viscosity and other measurements, see Bailey, (1925).

in which x = logarithm of concentration, y = logarithm of viscosity, and n = the number of observations, while b is the tangent of the angle which the plot forms with the axis of abscissa. This has come to be known as the "quality constant *b*" and in the opinion of Sharp and Gortner the numerical value of constant *b* will increase with an increase in the quality of the glutenin. When the value for *b* determined by Sharp and Gortner was inserted in the equation

$$\frac{(\text{glutenin } \%).\ (b)}{\text{loaf volume}}$$

an approach to a constant was encountered in the bread flours of patent grade that were milled from hard wheat. This would imply a linear relation between the constant *b* and loaf volume in the flours which have the same glutenin content but vary in baking strength, as evidenced by loaf volume. In a recent communication Blair, Watts, and Denham (1927) state that the plot of logarithm of viscosity against logarithm of concentration is not a straight line, but takes the form of a simple parabola. When the logarithm of viscosity is plotted against concentration, the resulting plot is a straight line and its slope may be used as a measure of flour strength.

The 17 flours included in these studies were subjected to viscosity determinations after the method described by Gortner (1924). The MacMichael viscosimeter was used and the constant *b* was calculated by the formula given above. When this value was multiplied by the glutenin content, Method I, and the product divided by loaf volume, the resulting values (Table V, seventh column) were not at all constant, but varied from 0.0039 to 0.0065. Nor can any relation be traced between the values thus computed and other properties of the flour. The coefficient of correlation of loaf volume with this quality constant *b* was 0.074 ± 0.163, r being less than the probable error and of such small magnitude as to be of no significance. It is, accordingly, concluded that so far as these samples are concerned, the quality constant *b* afforded no measure of flour strength.

Diastatic activity.—It is recognized that the diastatic activity of flour as measured in terms of the autolytic saccharogenesis, or rate of production of sugar from starch, in a simple flour suspension in water is significant in predicting the quality of bread that can be produced from flour.

Rumsey (1922) suggested a method for the estimation of diastatic activity which was used in the examination of these 17 flour

samples. In applying the Rumsey method, 10 grams of flour was digested with water at 27° C. for 1 hour, diastatic activity was inhibited at the end of the hour by acidulation, and the digestion mixture defecated and clarified with sodium tungstate solution. Reducing sugars in an aliquot were determined by the Quisumbing-Thomas method and computed as equivalents of maltose in milligrams per 10 grams of flour.

Rumsey reported on a number of flours which varied in diastatic activity from 34.8 mg. of maltose per 10 grams of flour to 308.1 mg. In general, there appeared to be a positive correlation between diastatic activity and baking strength, at least to the extent that flours which were markedly deficient in this property did not readily yield satisfactory bread.

Diastatic activity of the 17 flours included in this study ranged from about 90 ± mg. of maltose in the soft wheat flours from the Pacific Coast, Indiana, and Ohio (Samples Nos. 1, 7, 8, and 13), to 290± in the hard spring wheat flours, Nos. 3 and 10. The soft wheat flour, No. 15, milled from Missouri wheat, was higher in diastatic activity than the other soft wheat flours examined, which may account in part for the superior baking qualities of this flour, to which attention has already been called.

In an effort to ascertain the significance of diastatic activity, the following computation was made: diastatic activity in mg. per 10 grams of flour was multiplied by the crude protein content in percentage, which product may be designated as x. Then the loaf volume in cc. was multiplied by the texture score, yielding a value designated as y. The coefficient of correlation $r. = 0.384 \pm 0.080$. This is a significant positive correlation and indicates that diastatic activity probably plays an important rôle in determining the strength of flour as evidenced by the volume and texture of experimental loaves.

It is improbable that a partial correlation of diastatic activity and baking strength (with constant protein content) would be large, because observations have indicated that each increment of increase in diastatic activity above a certain level has little effect on baking strength. An equivalent increment of diastatic activity of flours initially deficient in that particular, registers a significant effect upon loaf volume and texture of the bread. This implies that diastatic activity is not a linear function of loaf volume, in which event the conventional computation of the coefficient of correlation yields results of limited value. The data available at

this time are not adequate to an extension of this discussion, and further work must be done before numerical values can be placed upon the relation between diastatic activity and baking strength.

The factors directly responsible for variations in diastatic activity of such material as wheat flour are not easily recognized. Whether saccharogenesis depends chiefly upon the concentration of the enzyme, the characteristics of the substratum, or the reaction of the medium, remains to be determined. It appears quite certain from the work to date that all these factors are involved and possibly others as well.

Starch granules and diastatic activity.—The studies of Whymper (1909) led to the conclusion that large starch granules are more readily attacked by diastases than small ones. Armstrong (1910) measured the diameter of starch granules of wheat flour and found that the small granules range in size from 3 μ to 5 μ and the largest from 30 μ to 35 μ. The larger granules constituted from 1.5% to 6% of the total number in the flour which Armstrong examined, which means that from 30% to 40% by weight of these granules are encountered in flours containing the largest percentage. Buchanan and Naudain (1923) concluded that strong flours have the largest percentage of small granules. They considered that the average size of all the granules present is more significant as a criterion of strength than the percentage of small or large granules.

The observations of Rask and Alsberg (1924) on the properties of starch and their relation to baking strength indicated that it might be advisable to give further attention to this constituent of the flours examined. Starch was prepared from each of the samples after the method of Rask and Alsberg (1924), except in the manner of drying the starch. These starch preparations were dried in an air oven for 12 hours at 75° C., then pulverized to a powder that would pass through a No. 8 xx flour silk, redried in an air oven at 100° for 12 hours, and finally in a vacuum oven at 100° for 6 hours.

In determining the relative size of starch granules, 1 mg. of starch was suspended in 100 cc. of water. A drop of this suspension was transferred to a ruled hemocytometer slide, a small quantity of a solution of iodine and potassium iodide was added, and the cover slip was placed under the ruled area. The compound microscope used in the examination of these starch granules was provided with an eye piece micrometer. Each division of the scale of this micrometer was equivalent to 3.7 μ. It was found conven-

ient to divide the starch granules into three groups or classes on the basis of size, namely,

1. those having a diameter of less than 7.4 μ,
2. those having a diameter of 7.4 μ to 14.8 μ,
3. those having a diameter of more than 14.8 μ.

About 700 granules were measured and classified in each sample. The percentage of each size of granule in terms of the number of granules measured in each of the 17 flour samples is recorded in Table VI. Using as a basis of comparison the percentage of small granules (less than 7.4 μ in diameter), there was considerable variation in the proportion of the different sizes in the several samples. No correlation could be discerned between this characteristic of the flours and their source or the type of wheat from which they were milled, nor was there any significant correlation between the size of starch granules and baking strength as evidenced by loaf volume. The coefficient of correlation of these two variables was 0.278 ± 0.144. When the coefficient of correlation between the size of starch granules and diastatic activity of the flour was computed, it was likewise found to be negligible, as $r = -0.020 \pm 0.168$. It accordingly appears that differences in the properties of starch that are related to baking strength of the flour or to diastatic activity alone must be attributable to properties of the starch other than those involved in the size of the granules alone.

TABLE VI

PERCENTAGE OF STARCH GRANULES OF DIFFERENT SIZES IN 17 FLOUR SAMPLES AND HEAT OF IMBIBITION OF DRY STARCH

Lab. No.	Diameter of starch granules			Heat of imbibition
	Less than 7.4 μ	7.4 μ-14.8 μ	Over 14.8 μ	
	%	%	%	°C.
1	80.45	6.62	12.93	11.3
2	86.36	4.63	8.99	10.5
3	59.97	13.26	25.76	10.1
4	75.03	9.97	15.00	12.8
5	85.92	3.92	10.17	12.6
6	79.84	6.00	14.15	11.2
7	76.65	3.64	19.70	12.0
8	88.16	3.50	8.33	12.8
9	77.94	7.35	14.70	11.1
10	79.07	6.86	14.06	12.5
11	83.01	5.01	11.97	11.5
12	89.55	3.67	6.78	11.6
13	87.78	4.57	7.65	12.2
14	91.79	2.67	5.53	11.8
15	82.80	5.29	11.90	11.5
16	74.39	8.61	17.00	13.0
17	87.77	4.04	8.18	11.7

This conclusion is further supported by observations made on the heat of imbibition of these starch preparations. This was de-

termined by placing 20 cc. of water at 22° C. in a small Dewar flask which was then placed in a water thermostat at 22° C. Twenty grams of the starch, previously dried as indicated above, was stirred into the water in the Dewar flask. The rise of temperature was noted with a high grade thermometer that could be read to 0.05° C. and is recorded in the last column of Table VI. The differences among the flours are substantial amounting to 2.5°, a range of from 10.5° C. to 13.0° C. No correlation could be discerned between the heat of imbition and the properties of starch revealed by the size of starch granules. Thus, the coefficient of correlation of heat of imbibition in degrees C. per 20 grams of starch with the percentage of starch granules less than 7.4 μ in diameter was -0.0004 ± 0.1686.

Summary

Flour samples representing straight grade or patent flours milled from hard spring, hard winter, soft red winter, and Pacific Coast white wheats were collected from various parts of the United States.

Glutenin content was determined by the Sharp and Gortner method and also by the Blish and Sandstedt method and the results obtained were in satisfactory agreement.

Ratio of glutenin to crude protein and of glutenin to the sum of glutenin and gliadin was computed in all the flours and no significant variation was detected.

Baking tests conducted by three different methods failed to arrange the flours in the same order as judged by loaf volume.

Coefficient of correlation of the percentage of crude protein with the loaf volume of bread baked after the method suggested by the Committee on Standard Formula and Method of Procedure for Experimental Baking Tests, of the American Association of Cereal Chemists (1925), was 0.678 ± 0.091.

Extensibility of doughs prepared from these flours and tested with the Chopin extensimeter varied appreciably.

Coefficient of correlation of extensibility and loaf volume was 0.671 ± 0.076.

Constant *b* computed from viscosity determinations involving several concentrations of leached flour acidulated with lactic acid was not correlated with the results of the baking tests.

Diastatic activity of the flours ranged from the production of $90 \pm$ mg. of maltose per 10 grams of flour in one hour at 27° C. to $290 \pm$ mg. of maltose.

Coefficient of correlation of diastatic activity with loaf volume was 0.384 ± 0.080.

Proportion of large starch granules (over 14.8 μ), small starch granules (less than 7.4 μ), and intermediate starch granules (7.4 μ to 14.8 μ), was determined by counting the number of each size in starch suspensions mounted in a ruled hemocytometer slide.

No correlation of this characteristic of the starch and the results of baking tests was evident, nor was there any correlation between size of starch granules and diastatic activity of the flour.

Heat of imbibition of the dried starch preparations was not correlated with any of the other properties of the starch or of the flour that were measured in these studies.

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